Tappi

THE JOURNAL OF THE TECHNICAL ASSOCIATION OF THE PULP AND PAPER INDUSTRY

OCTOBER 1961

VOLUME 44 • NUMBER 10

REC'D OCT 18 1961

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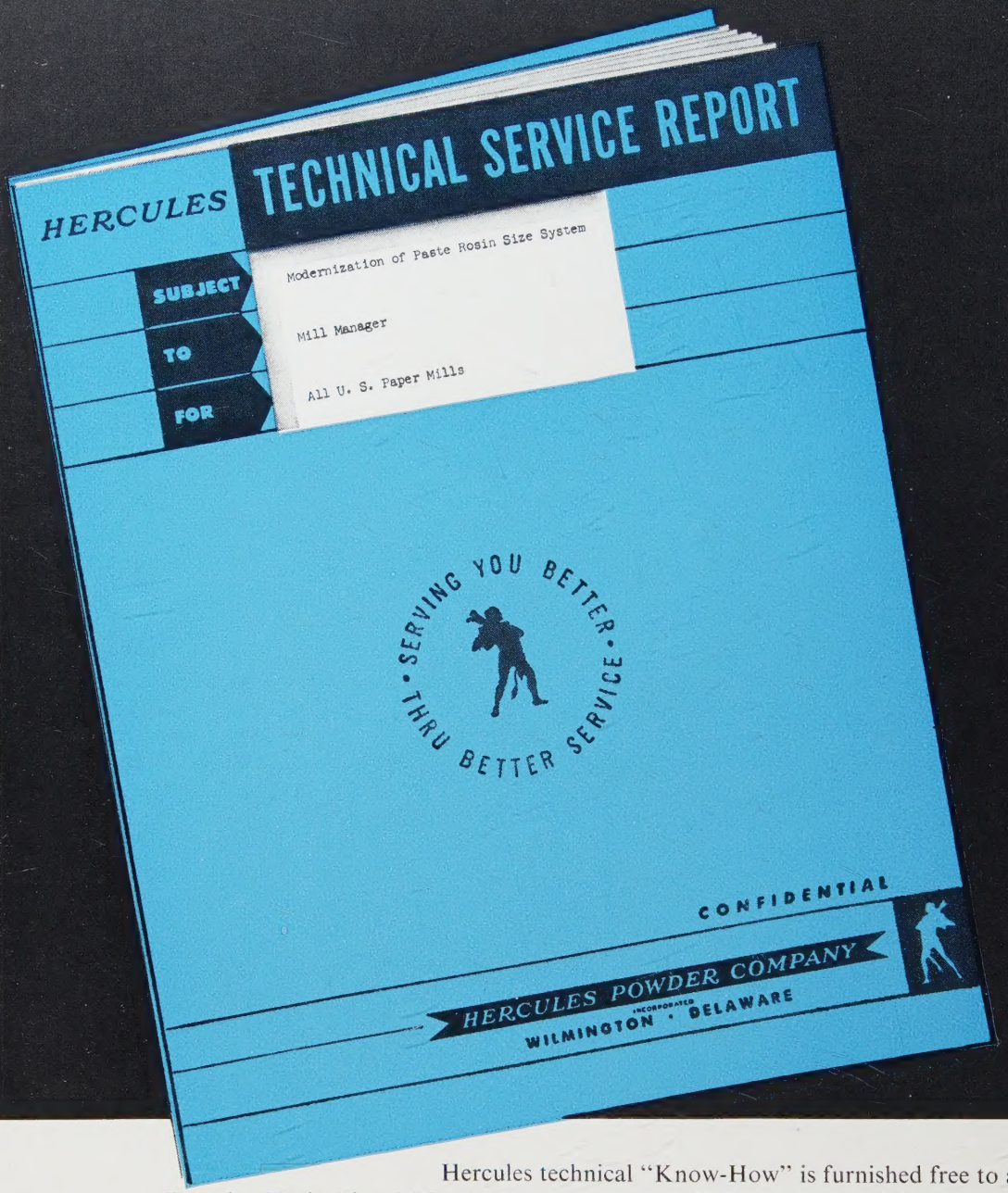

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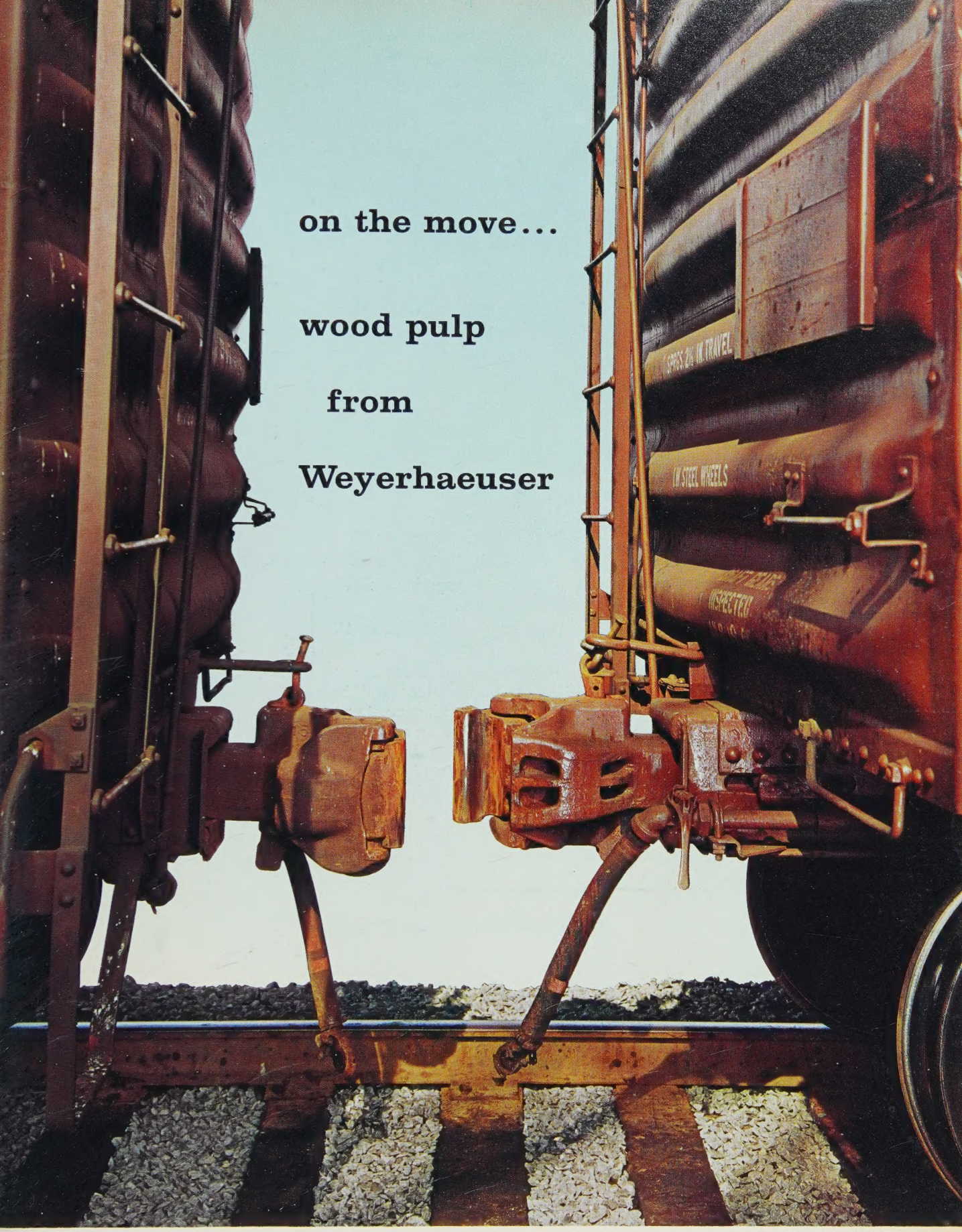

PHOTOGRAPHY BY GEORGE NORDHAUSEN

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DICALITE®
TECHNICAL REPORT
DICALITE DEPARTMENT • GREAT LAKES CARBON CORPORATION • 612 SO. FLOWER ST., LOS ANGELES 17, CALIFORNIA

New Dicalite BULK-AID for Paper Products

TRADEMARK

Dicalite BULK-AID is a new development in inorganic fillers, with particle size range and distribution carefully controlled to give maximum bulking characteristics in paperboard and paper products. BULK-AID has the lowest apparent or bulk density of any known mineral filler for papermaking use, thus providing required caliper at lower weights. One example is that of a 40-point groundwood sheet whose weight was reduced from 29.2 lbs/ft^3 to 27.3 lbs/ft^3 by the addition of 10% BULK-AID.

The light weight and bulking characteristics of BULK-AID make it especially suitable for use in paperboard; its high brightness and excellent retention are also of considerable value. BULK-AID lowers the furnish cost and increases pulp mileage in top liners for patent white paperboards. Its brightness and opacity raise the quality of underliners at the same time it extends the furnish.

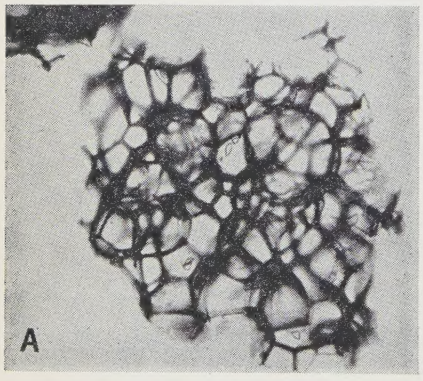
Dicalite BULK-AID is an amorphous mineral, technically an aluminum silicate, produced from a volcanic mineral known as perlite by a special process developed by Great Lakes Carbon Corporation. BULK-AID is inert, insoluble in water, acid or mild alkalies. It is grit-free, and exhibits no reaction with alum in sizing or with other papermaking chemicals. It is readily dispersed in any furnish, does not cause foam, and will not fill felts.

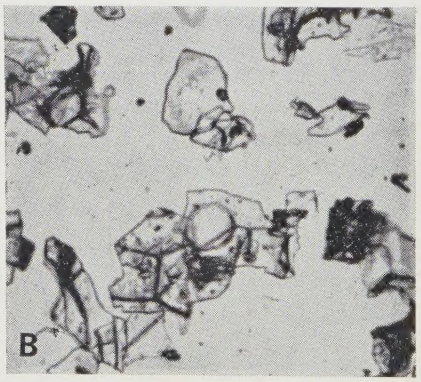
Among BULK-AID's physical characteristics are these:

Color: White
Average G.E. Brightness: 83
Loose weight: 4½ lbs/ft^3 average
Bulk densities in paperboard: 15 lbs/ft^3 average.
Particle size: 80% less than 10 microns

Extensive testing has shown BULK-AID to be successful in actual paper mill production. Most of these tests were on cylinder machines for paperboard production, but limited tests indicate its value in many slow speed fourdrinier applications.

Your Dicalite engineer will be glad to discuss BULK-AID with you and to advise on possible product applications in your own plant.





When perlite is crushed and then suddenly heated to the proper point in its softening range, it pops like corn into particles like that shown in photomicrograph A, containing a myriad of microscopic sealed cells. The size of the original particle and the application of heat are both important, and are critically controlled. This is the expanded material from which BULK-AID is produced. Controlled milling and air classification by an exclusive Great Lakes Carbon Process are the major steps in making BULK-AID, typical particles of which are shown in photomicrograph B.

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Gordon G. Halvorsen

DICALITE'S "MAN ON THE SPOT"

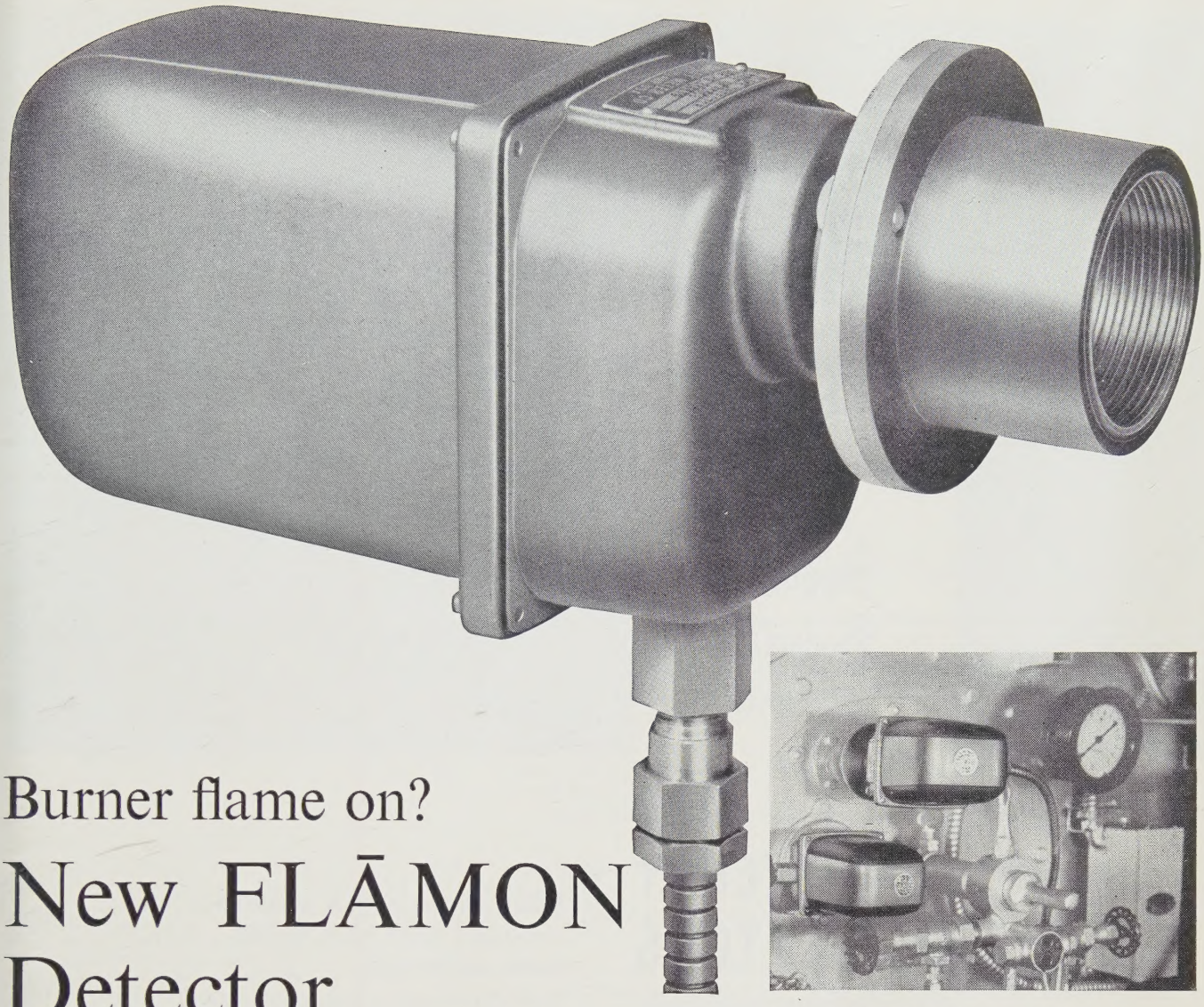
The development of Bulk-Aid, to meet the industry's need for an extremely low density bulking agent, was initiated by Gordon Halvorsen, Product Sales Manager for Dicalite, and he has been closely identified with its tests and product application work from the beginning.

Halvorsen has an extensive background in research and development work, beginning with his graduation from the University of Minnesota with a degree in Chemical Engineering. Prior to joining Great Lakes Carbon in 1940, Gordon had been engaged in technical and laboratory work in the pharmaceutical, corn processing and water treatment fields, and continued his research work in the Dicalite product laboratories for the first 5 years of his association.

Halvorsen is the author of several technical papers, and has contributed to both paper industry and brewing publications. Among his technical society memberships he lists TAPPI, American Society of Sugar Beet Technologists, Master Brewers Association of America, and others in the brewing and drycleaning fields.

NEW DATA SHEET

on Dicalite *BULK-AID* gives more complete information on physical and chemical properties, and on its advantages in papermaking. Write for your copy to Dicalite Dept., 612 So. Flower St., Los Angeles 17, Calif.



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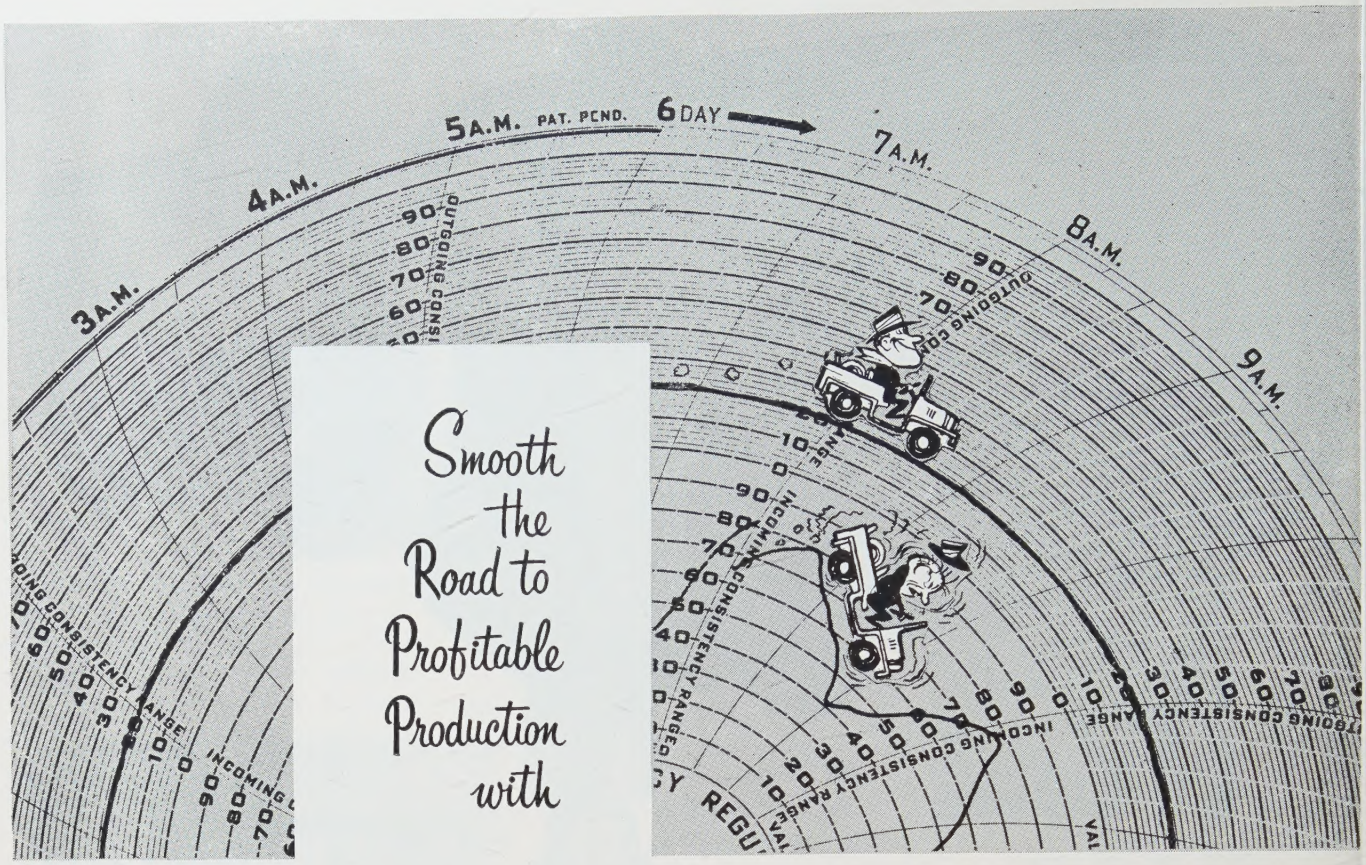

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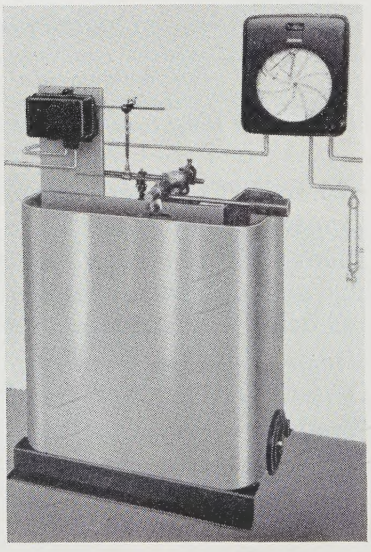

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National Conferences

Fifteenth Alkaline Pulping Conference
Rice Hotel, Houston, Tex.
Nov. 1–3, 1961

Forty-seventh Annual Meeting
Commodore Hotel, New York, N. Y.
Feb. 18–22, 1961

Thirteenth Coating Conference
Netherland-Hilton Hotel,
Cincinnati, Ohio
May 14–16, 1962

Thirteenth Testing Conference
Benjamin Franklin Hotel,
Philadelphia, Pa.
Sept. 18–20, 1962

Seventeenth Plastics-Paper Conference
Statler-Hilton Hotel, Boston, Mass.
Oct. 8–11, 1962

Seventeenth Engineering Conference
Queen Elizabeth Hotel, Montreal, Que.
Oct. 14–19, 1962

Twelfth Corrugated Containers Conference
Statler-Hilton Hotel, Boston, Mass.
Oct. 23–26, 1962

Sixteenth Alkaline Pulping Conference
DeSoto Hotel, Savannah, Ga.
Oct. 31, Nov. 1–2, 1962

Please refer to Local Section Activities for the locations and dates of Section Meetings

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For over 36 years, Industry has looked to West End Chemical Company as a dependable source of chemicals. Soda ash, sodium sulfate, salt cake, quicklime, hydrated lime and borax are included in the product line.

Located in the Mojave Desert some 190 miles northeast of Los Angeles, West End's Searles Lake location has long been recognized as one of the world's largest sources of inorganic salts. Searles Lake, which is now dry, was formed through the ages by waters carrying small quantities of the various salts in solution from the Sierra mountain range to what was then an inland sea. Subsequent volcanic activity blocked off the flow of these waters, leaving the lake to evaporate to its present day level. These acts of nature caused enormous quantities of inorganic salts to be left in the Searles Lake Basin.

West End's connection with Searles Lake dates back to that period following World War I when numerous companies were experimenting with various processes for extracting mineral wealth. After a number of years of experiment and research, the Hellmers Process (H. D. Hellmers, now West End Vice-President, Production) was adopted, and it is still the basis for today's operations.

Briefly described, the Hellmers Process involves the pumping of brine from the lake, treating the brine with carbon dioxide gas to form sodium bicarbonate which precipitates out. In the carbonation phase of the process, the borates contained in the brine as sodium metaborate and sodium tetraborate are converted to the more acid higher borates of sodium by the reaction of CO_2, eliminating part of the Na_2O by forming $NaHCO_3$. After separation of sodium bicarbonate from the carbonated brine, the latter is mixed with additional untreated brine from the lake in such proportions that practically all of the borates in the mixture are in the form of sodium tetraborate. This results in a state of supersaturation with respect to sodium tetraborate, which is crystallized from the mixture by the simple process of cooling and agitating. After settling, the brine is sent to the sodium sulfate recovery units and the settled borax recovered and refined. The bicarbonate recovered in the first step of the process is filtered, washed free of impurities, and converted to various grades of soda ash.

For a number of years soda ash and borax were West End's sole products. In 1938, installation of a hydrator made it possible to market hydrated lime.

In 1955, encouraged by the growth of the kraft pulp and detergent industries, West End made its first offering of salt cake and anhydrous sodium sulfate. In the intervening years, West End has become a major national source of these chemicals.

The new West End soda ash plant now under construction at a second natural source in Wyoming is scheduled on-stream in 1962. Produced under the same uniform management and controls of the parent plant, high quality West End soda ash from Wyoming will be available to industry in an ever-broadening market.

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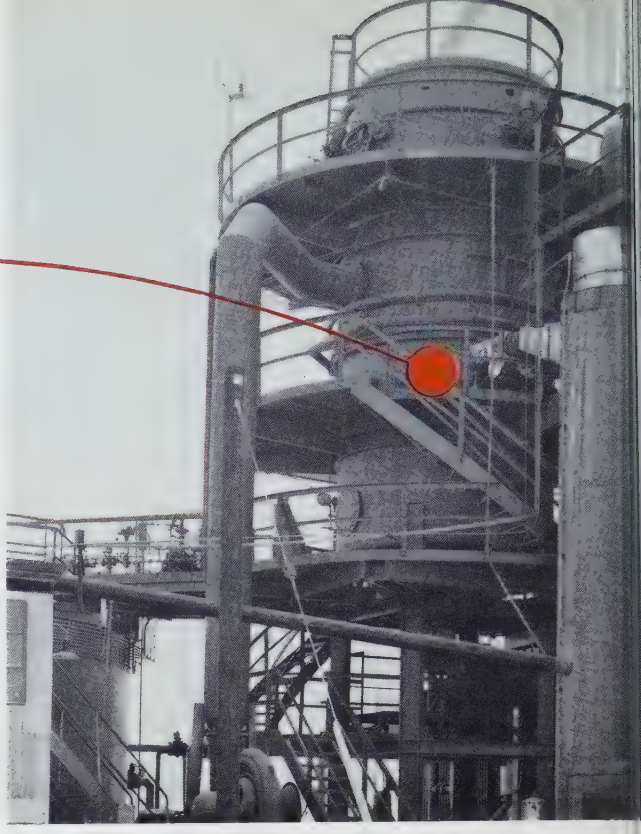
WEST END SALT CAKE



Since mid-1955, West End has been recognized as a major producer of high-quality salt cake. Original production capacity of salt cake at the Westend plant was 50,000 tons annually, but with completion of a second unit in the fall of 1956, production capacity was increased to 120,000 tons per year. With completion of a third unit in 1961, capacity will increase to 200,000 tons per year. Today, West End is solidly qualified to handle the complete requirements of its customers dependably, economically and efficiently. West End salt cake is being used to a wide extent in kraft paper mills in the Pacific Northwest and the Southeastern section of the United States wherever a sulfate pulp process is utilized.

SALT CAKE

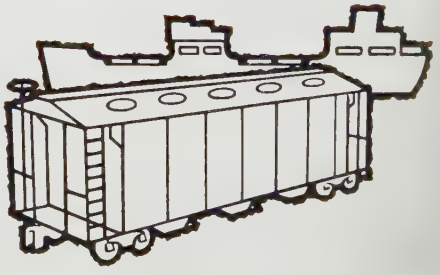
TYPICAL ANALYSIS

	Typical %	Guarantee %
Na_2SO_4	99.57 -99.87	99.5 Min.
NaCl	.06 -.14	.2 Max.
B_2O_3	.03 -.06	.1 Max.
CO_2	.02 -.04	.05 Max.
Na_2O	.05 -.09	.1 Max.
H_2O	.01 -.10	.2 Max.
Insoluble	.001-.01	.05 Max.

Density:
Struck Approx. 75 lbs./cu.ft.
Tapped Approx. 88 lbs./cu.ft.

Solution . . . . . Clear
Color . . . . . White

WEST END SALT CAKE IS MOVED ECONOMICALLY at great distances by truck, ocean vessel or by West End's own fleet of leased covered hopper rail cars. Stauffer of Canada storage depot . . . The West End project that solved the problems of time and cost of other means of transportation . . . typifies West End advance planning that provides customers with the benefits of reduced inventory requirements, lower transportation costs and prompt delivery.



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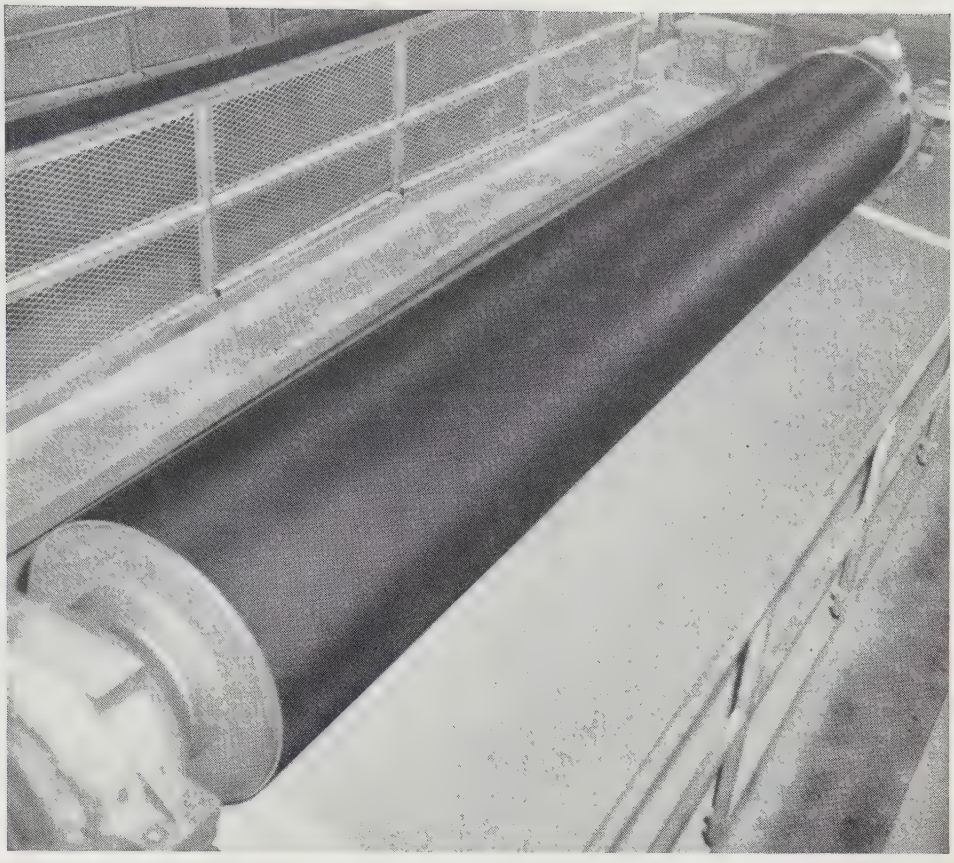

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Incidental Additives to Food: Have We Made a Prudent Judgment?

RICHARD C. NELSON

The history of the Food Additives Amendment of 1958 is reviewed briefly with special emphasis on the regulations pertaining to incidental additives. The problems of analysis are considered. The legality of "Blanket Regulations" is questioned as is factory inspection. On the basis of present experience with the existing law and its interpretation, it is suggested that Congress be requested to review it and the problems it has created.

IT HAS been said that men should strive for "prudence to decide what things should be feared, when they should be feared, and how much; and so a prudent judgment is involved in fearing the right things at the right time and in the right manner—neither too much nor too little."[1]

Nearly three years ago Congress, at the behest of the Food and Drug Administration, judged that a group of chemical substances, which we will call "incidental food additives," should be regulated in the same manner as "direct additives." That judgment, expressed in the Food Additives Amendment of 1958,[2] already has resulted in the expenditure of several tens of millions of dollars, of a vast amount of precious scientific and laboratory time, and of an even greater amount of less precious, but still costly, executive, administrative, and legal effort—private and governmental. This paper will consider the prudence of that Congressional judgment, at least insofar as one type of incidental additive is concerned.

The first question is not whether there is some form of control which might be desirable for incidental additives, but rather whether the present form of control s suitable. "Prudence" means the "provident use of resources."[3] If we are using our limited research facilities (and spending the consumer's dollar) improvidently because of an inapt statutory plan, we are being imprudent. If one of our finest regulatory agencies is drifting toward unauthorized and improper forms of regulation because of an inapt statutory plan, we are injuring our entire governmental fabric. Is the Food Additives Amendment causing these results? Is it an imprudent form of regulation for some types of incidental additives?

DEFINITIONS

The terms "incidental[4] food additive" and "direct food additive" do not appear in the statute. They are pragmatic classifications of chemicals which fall within the statutory definition of "food additive."[5] In dealing with "food additives" within that statutory definition, the "incidental additives" are those which are used to accomplish some mechanical, chemical or physical purpose *other* than in the food. Conversely, "direct additives" are those intentionally placed in food to achieve a desired result in the food item itself.

There are many sources of incidental food additives.[6] However, incidental additives from food packaging materials have proved to be more difficult to subject to the present statutory procedure than other types of incidental additives and are an excellent example of the problems encountered. This paper will be, for the most part, restricted to incidental packaging additives.

UNEXPECTED MAGNITUDE OF INCIDENTAL ADDITIVE PROBLEM

The voluminous testimony during the Congressional hearings which preceded enactment of the Food Additives Amendment did not indicate that incidental additives were a major or even a substantial hazard to health. The testimony was almost entirely directed toward direct additives. Incidental additives seem to have been included in the coverage of the Amendment primarily because the Food and Drug Administration felt that the Amendment should make no distinctions as to types or sources of additives, and that it would be in the public interest to impose the same pretesting requirements on all sources of chemicals in food.

We find, however, that the bulk of the FDA's time and effort in the additive field is being devoted to incidental, not direct, additives.[7] Packaging additives, particularly, have absorbed a great deal of effort, not because anything of concern was discovered, but simply because the number and complexity of this type of

RICHARD C. NELSON, Crown Zellerbach Corp., San Francisco, Calif.

[1] Quoted from "The Great Ideas" by Kenneth E. Mulford, *FDCLJ* 16, No. 2: 117 (February, 1961).

[2] Public Law 85-929, Sept. 6, 1958.

[3] Webster's New International Dictionary, Second Edition.

[4] The words "indirect" and "unintentional" are used in the same sense.

[5] Section 201(s) of the Federal Food, Drug and Cosmetic Act, 21 U.S.C. 321(s), reads as follows: "The term 'food additive' means any substance the intended use of which results or may reasonably be expected to result, directly or indirectly, in its becoming a component or otherwise affecting the characteristic of any food (including any substance intended for use in producing, manufacturing, packing, processing, preparing, treating, packaging, transporting, or holding foods; and including any source of radiation ntended for any such use), if such substance is not generally recognized, among experts qualified by scientific training and experience to evaluate its safety, as having been adequately shown through scientific procedures (or, in the case of a substance used in food prior to January 1, 1958, either scientific procedures or experience based on common use in food) to be safe under the conditions of its intended use; except that such term does not include: (1) a pesticide chemical in or on a raw agricultural commodity; or (2) a pesticide chemical to the extent that it is intended for use or is used in the production, storage or transportation of any raw agricultural commodity; or (3) a color additive; or (4) any substance used in accordance with a sanction or approval granted prior to September 6, 1958 pursuant to this chapter, the Poultry Products Inspection Act or the Meat Inspection Act of March 4, 1907, as amended and extended."

[6] The main sources of incidental additives are: agricultural chemicals (e.g. a pesticide which leaves some residue in a finished food); animal medications (e.g. a drug which leaves a residue in milk, eggs or meat); processing chemicals (e.g. a chemical residue contributed by processing or by conveying equipment); and packaging materials (e.g. polyethylene film when a chemical component migrates).

[7] "It is . . . the indirect additives which have been responsible for the major part of the initial confusion arising in the field, and which have absorbed a major part of the effort and time of both the administrative agencies and industry during the past year." Dr. Kenneth Morgareidge, Food and Drug Research Laboratories, *FDCLJ* 15, No. 10: 672 (October, 1960).

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incidental additive were far beyond anyone's expectation. As the General Counsel for Food and Drugs put it:

> "The magnitude of this problem is far greater than ever anticipated. Instead of several hundred additives we thought would need clearance, there are a few thousand and seemingly, no one can sell any package or any processing equipment or the materials going into them, unless FDA has first cleared the substance for safety."[8]

Originally, FDA estimated that by March 6, 1960, 800 to 1000 final Regulations would have been issued.[9] The slow progress toward final Regulations, and the significance of incidental additives to the rate of progress, appear from this quotation, made over a year after the March 6, 1960 date:

> "Since the enactment of this law in September, 1958, the Food and Drug Administration has responded to more than 4200 formal inquiries dealing with food additive problems . . . there have been hundreds of discussions with industry people . . . we have received 391 petitions for food additive regulations . . . *175 were for indirect additives involving approximately 1675 chemicals;* 216 were for direct additives involving approximately 257 chemicals . . . *we have issued 59 regulations to date* . . . we have issued extensions of the effective date of the law covering some 3000 uses of food additives, about 1100 (of) which are direct additives. The others[10] are in the packaging and equipment field . . . [11] (Emphasis supplied.)

UNIQUE PROBLEMS OF PACKAGING ADDITIVES

Why is the problem of obtaining a Food Additive Regulation more difficult where packaging additives are involved than where direct additives are the concern? The Amendment was written in terms of "any substance" and its procedures clearly are designed to deal with individual chemicals, not end products. The law is fundamentally unsuitable, in its present form, to a large number of chemical substances which are potential additives, grouped together in widely varying amounts within a single product.

Although its views may have changed, the Food and Drug Administration made it clear in the early days of the Amendment that all potential additives must be considered individually:

> "As we see it, there are two fundamental principles which may well have been overlooked by some in the packaging industry: The first of these is the matter of just what we should be talking about when we deal with the food-additives question. Certainly, initially we are not dealing just with the finished package. Instead, we are concerned with such of its individual components . . . which may reasonably be expected to migrate to the food. As a basic premise, therefore, it is of paramount importance to learn first just what components go into the package . . ."[12]
>
> "In seeking a solution to this problem we need, first, the complete quantitative composition of the film or coating . . . and, secondly, data showing with the necessary degree of sensitivity and reliability the quantity of each component which may transfer to the food . . . Since a change in the percentage of one component of a plastic, or in the manufacturing process . . . may have a marked effect on the chemical and physical properties of the plastic, we are constrained to recommend that each formulated packaging material be individually evaluated."[13]

[8] W. W. Goodrich, *FDCLJ* 16, No. 2: 98 (February, 1961).
[9] Deputy Commissioner Harvey, *FDCLJ* 15, No. 5: 306 (May, 1960).
[10] There would be 1900 others.
[11] Assistant FDA Commissioner Kirk, *FDCLJ* Vol. 16, No. 5, at pp. 284–285 (May, 1961).
[12] Assistant to the FDA Commissioner J. Kenneth Kirk, *FDCLJ* 15, No. 4: 264 (April, 1960).
[13] L. L. Ramsey, FDA Division of Food, *FDCLJ* 13, No. 12: 789 (December, 1958).

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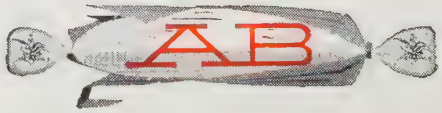

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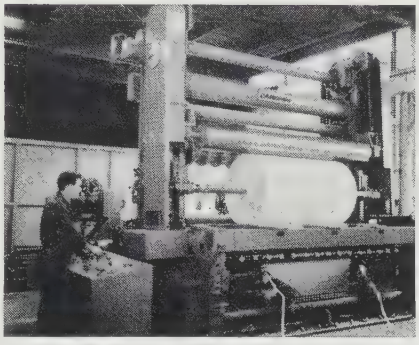

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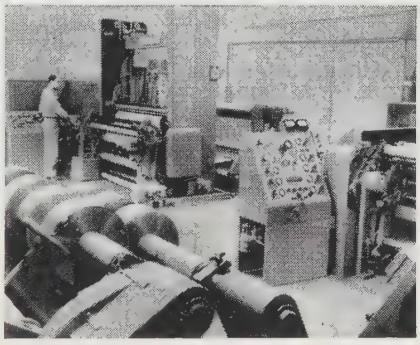

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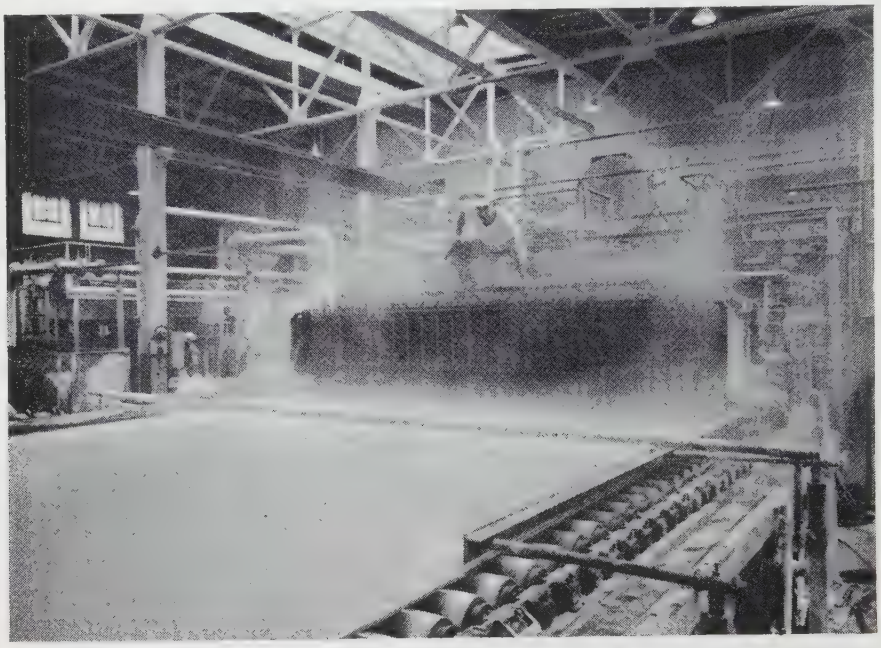
Inevitably, the practical problems of applying an individual component approach to highly complex end products (such as cellophane, paper, can enamels, etc.) began to appear. The major problem was the difficulty of determining the identity and quantity of each migrant. This problem normally does not face the proponent of a direct additive because he knows right from the start the identity and purity of his additive, and the quantity he proposes to introduce into the food. He has plenty of problems, to be sure, for he must determine the safety of his direct additive and this may require animal feeding tests costing hundreds of thousands of dollars and requiring several years of study. The proponent of an incidental packaging additive, however, may never be able to reach the stage of animal feeding tests because of the difficulties involved in analyzing for migrants from packaging materials. For him the question is not only whether there is enough time and money to do the job, but also whether it is scientifically possible to do it at all.

THE ANALYTICAL PROBLEM

The statute says FDA must know what—if anything—migrates to the food, and how much. Consider, for example, a packaging material (e.g., paper) where the make-up sheet for the primary product shows 20 to 30 items. Some of these items are closely related to each other (e.g., possible residual calcium from the sulfite pulping process and calcium stearate, a lubricant in starch coatings). Some items are closely related to subsequent converting chemicals which will be used (e.g., urea formaldehyde resin, used to provide wet strength in primary papermaking, and formaldehyde which might be present in adhesives or coatings). Still other chemical items on the make-up sheet are proprietary mixtures known only by trade names (e.g., "Dowicide G," a slimicide, and "Nalco 71-K," a defoamer) and the composition of which may be trade secrets, if, indeed, the manufacturer knows the chemical composition with any exactness. Assume that this complex formulation of 20 or 30 items is sold for conversion into a specialty food paper (e.g., for wrapped bread) and in the conversion process is printed. (Inks are probably the most highly complicated mixtures of chemicals used in industrial processing; even a small ink maker may handle as many as 50,000 formulations a year.[14] In further conversion the printed paper product may be coated with a blend of several waxes, to which polyethylene may have been added. Rolls of the converted product are then shipped to food packers (e.g., bakers) who pass them over various types of wrapping equipment for the packaging of many varieties of the food (bread) at various temperatures, with the packaged food then being transported and held under varying conditions of temperature and moisture for varying lengths of time.

If we remember that the applicability of the Federal Food Additives Amendment to each one of the multitude of chemicals involved in this converted packaging product depends on whether even one molecule of one of these chemicals transfers to the packaged food under any one of

[14] "Toxicity and Printing Ink," National Printing Ink Research Institute, Lehigh University, Bethlehem, Pa.



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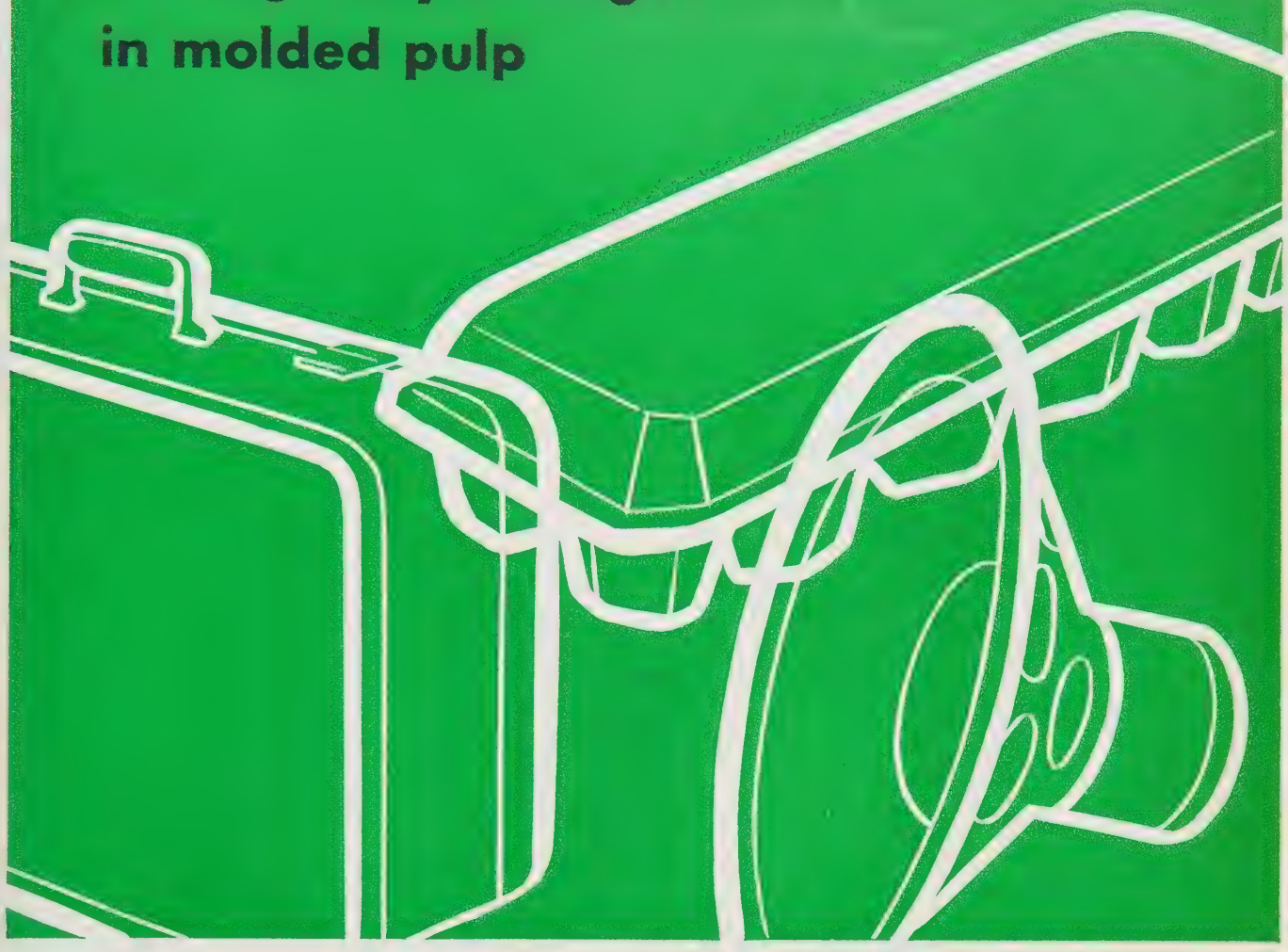

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the intended conditions of use, the enormous magnitude and the staggering complexity of the initial analytical problem can be grasped. However, for the sake of the example let us make a number of very unlikely assumptions. Number 1: let us assume that infinite time, money, and laboratory skill and facilities are available. Number 2: let us assume the manufacturers of the proprietary mixtures and the inks readily can and will reveal the exact formulation of their products, either to our laboratory or to the FDA. Tests are then conducted for the presence in the food of each chemical known to have been a possible constituent of the primary product and each chemical used in the converting process. Number 3: assume that where no analytical test is known to science, our hypothetical laboratory can promptly devise one. Thus each migrant and the quantity thereof becomes known.

The problems are only beginning, however. Each migrating chemical not only must be identified but its source should be known as well if it will be necessary to put any sort of limitation on its quantity. Many foods in their natural states contain substances identical with, or not capable of being distinguished from, substances which may become incidental additives. If our scientists find some fraction of a hydrocarbon in the bread, where could it have come from? The wax? Yes. From the polyethylene? Yes. From the defoamer which might have had some mineral oil in it? Yes. From the bread itself or from some direct additive to bread? Yes. Can the true source be determined? Probably not in the present state of scientific knowledge, but—back to the laboratory. Assumption no. 4: sources can be pinpointed.

There are still more variables. What migrates and how much may depend on the length of time there is a contact between the packaging material (the bread-wrap) and the food (bread). Not just length of time affects this determination, however; temperature, moisture, rough handling, and other factors are important. And are all breads themselves of the same chemical composition? Obviously not. Assumption no. 5: it is possible to establish all possible conditions of use and to evaluate their effect upon migration accurately.

Thus in the end every migrant (and its source) is identified, the quantity transferring is determined, and (assumption no. 6) the safety of all the migrating chemicals, in those quantities, under all conditions of use, is established.[15] Our hypothetical scientists heave a sigh of relief and prepare to return to what they may consider to be more constructive endeavors. Then the manufacturer who employs them, and who has spent hundreds of thousands of dollars achieving the matters covered by our six assumptions, asks if the same converted paper product can be used for wrapping meat, candy bars, cookies, breakfast foods, and ten other general categories of food. Are the same analytical tests which were devised for the bread applicable

[15] As previously stated, testing one chemical of unknown toxicity for safety may require several years of animal feeding tests and cost up to $500,000. The chemicals used in packaging materials are chosen for their ability to accomplish some physical effect in the package and not to make some direct contribution to the food. Thus there is less likelihood of a history of food use, or of prior animal tests, and a greater probability of having to conduct animal tests under the new law. However, since we are hypothesizing, we can dispose of this most difficult aspect of the problem with only a mention.

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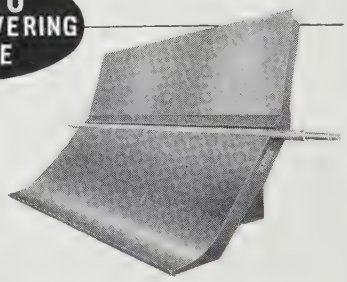

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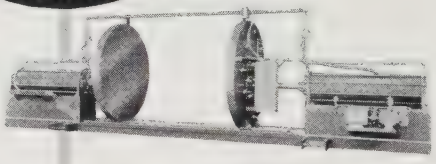

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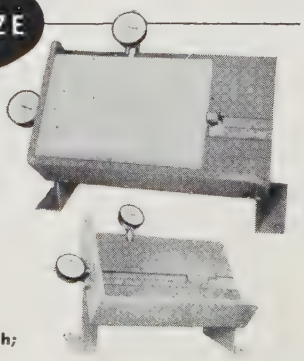

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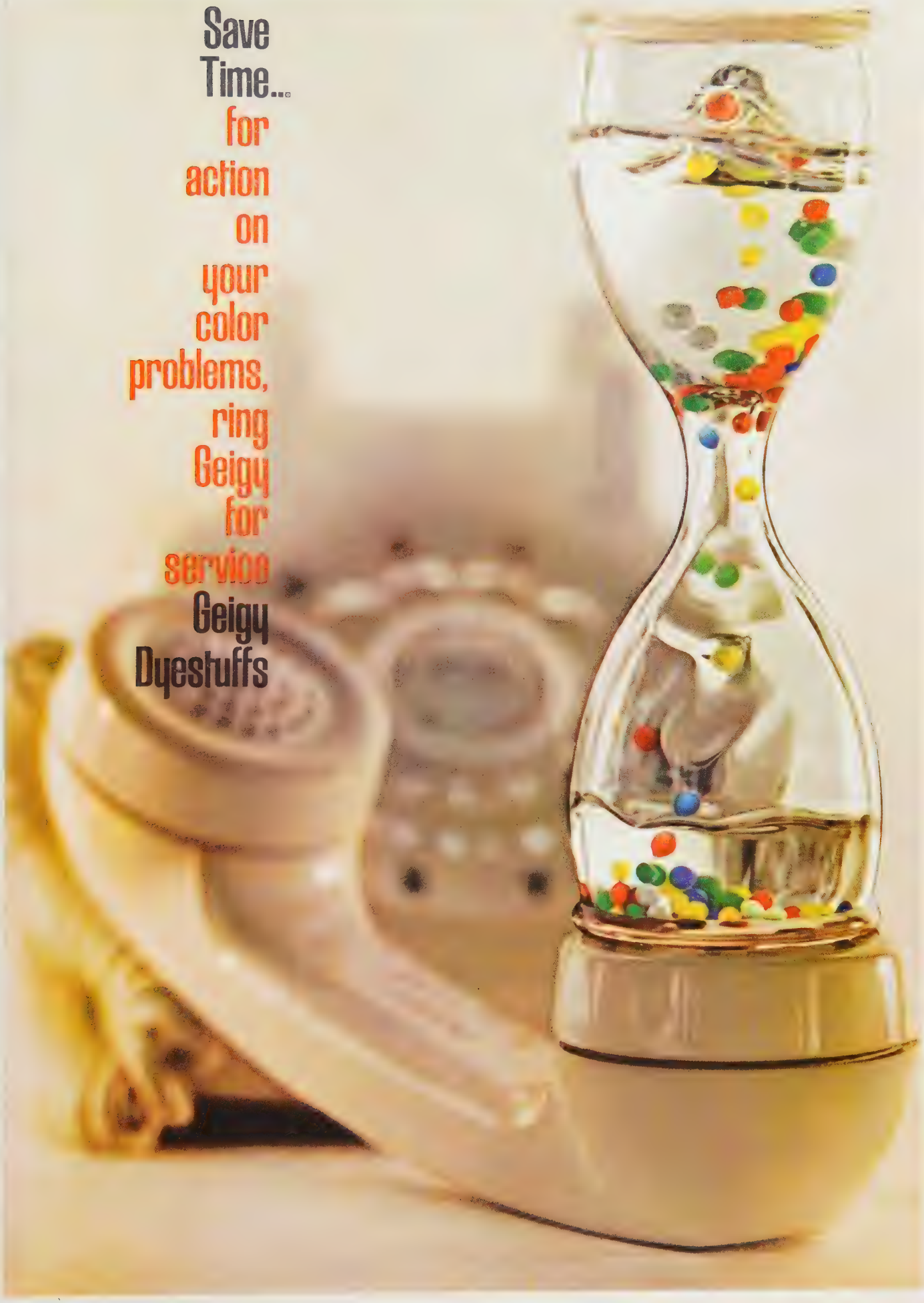
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to meat? Or to candy, etc.? Unfortunately not. Are the packing conditions, storage conditions, handling conditions, and time of contact the same? No. Back to the laboratory.

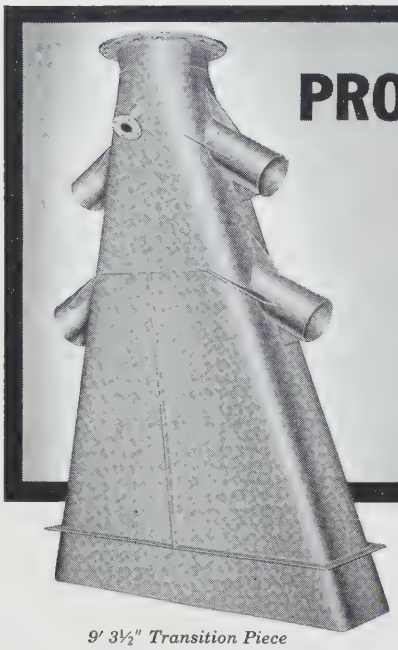
In the paper field alone, and without reference to the infinite varieties of converted products, a single manufacturer may produce more than 100 grades of primary food papers. Each grade involves several chemical differences from each other grade. Considering the variety of foods with which each grade may be used, and the variety of processing, transportation and storage conditions occurring in the case of different foods, the additive possibilities which the analytical chemist must consider are astronomical in number.

However, our weary scientists in our hypothetical laboratory are far from finished with their work when they have established the safety of all possible migrating chemicals, from all these products, to all possible packaged foods, under all possible conditions of use. Before a Petition can be filed obtaining a Regulation for any nonexempt chemical which is found to transfer to a food, under some possible condition of use, these scientists must develop not just an analytical technique which in their own laboratory will detect the quantity of the chemical present in the food, they must also devise a "*practical method* to determine the amount of the food additive in the . . . finished food."[16] (Emphasis supplied.) This does not mean a detection method requiring special equipment, knowledge or capability; what is envisioned is a method that can be used for practical food control purposes by any field laboratory of the Government.

Here we probably should stop making assumptions. The requirement of such "practical" analytical methods in circumstances such as have been described is, of course, highly unrealistic. It is perhaps not beyond the range of our imagination to assume that the finest scientists in the finest laboratories can perform wonders of analytical detection given infinite time and money. It is beyond reason to think that they can translate the brilliant results of specialized laboratory research into something akin to litmus paper tests for thousands of different chemicals under thousands of different use conditions.

The foregoing example, of course, is exaggerated to make its point. It takes no account of use of the "Ramsey solvents" which in some —but far from all—cases are enough like the food and the conditions of use to provide a satisfactory substitute for actual tests of the food. The example does not consider that in some cases a coating can be proved to be a complete barrier, thus eliminating the necessity for considering any ingredients except those in the coating itself. The example ignores the fact that in some end products (such as paper) a great many of the components are exempt from the law because of general recognition of their safety under the intended conditions of use ("GRAS"), or because they have prior sanctions,[17] in which case transfer of these substances becomes irrelevant. These helpful possibilities may lessen the magnitude of the problem, but they do not constitute a solution to it, or even offer sufficient assistance to make the law really workable. An additive pretesting law which is written in terms

[16] Section 409(b) (2) (D) of the Act, 21 U.S.C. 348(b) (2) (D), and 21 C.F.R. 121.51(e), D.
[17] See Note 5 supra.



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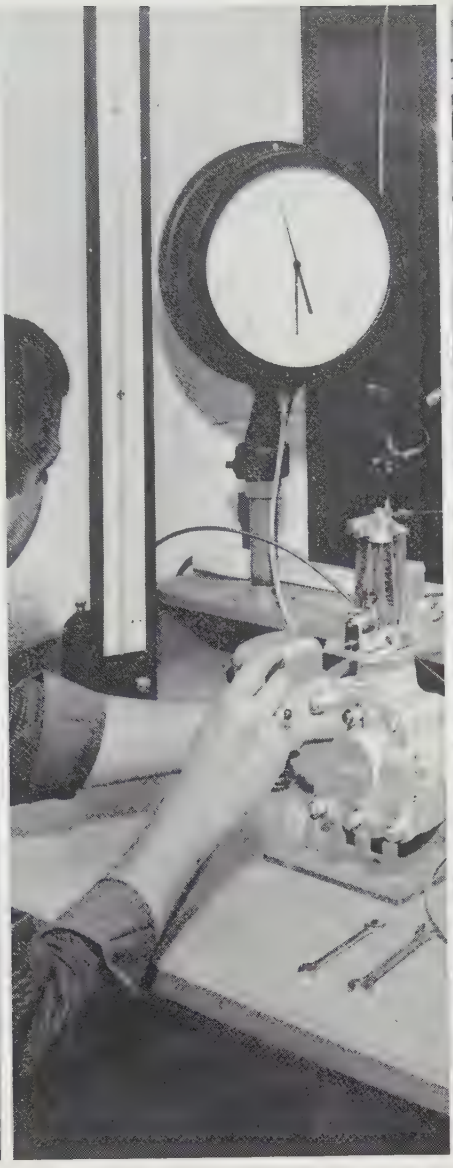

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of the migration of any quantity, no matter how small, of individual ingredients simply is not suitable for packaging materials. It seems the Food & Drug Administration has finally come to admit this:

> "Our original concept of a Food Additive Regulation was that this would specify the substance, state the purpose of its use and limit the amount which would be present in the food, and that we would, in every case, have a good method of determining whether this limitation has been met through examination of the finished food product in the laboratory . . . When we came to the packaging field, we were faced with the problem where it was just impossible to anticipate the development of methods of analysis which would enable us to examine food products and determine how much of the packaging material had migrated . . . Also in the packaging field our first concept was a consideration of the food additive status of each substance used in making a product such as paper, for example. This would work out on the basis that if the specific substance migrated to the food we would then have a Regulation for that specific substance . . . *it will, I believe, be quite apparent that approaching the problem on that basis would result in a monumental and perhaps never-ending job.*"[18] (Emphasis supplied.)

ADMINISTRATIVE LEGISLATION—THE "NEW" REGULATIONS

When it became apparent that the Food Additives Amendment could not be applied to incidental additives such as packaging materials according to its terms, the Food and Drug Administration had two choices. It could return to Congress with the frank admission that the incidental additive problem had been misjudged and ask Congress to reconsider the problem; or it could develop its own methods for handling such additives and "amend" the statute by administrative interpretations. The Administration chose the latter course and came up with a number of suggested procedures for short-cutting this "monumental and perhaps never-ending job."

FDA clearly has a duty to find a way of making the present statutory plan work efficiently for the consumer (and "efficiently" means that the cost to industry—and thus to the consumer—must represent value received by the consumer in terms of safety); or, if that is not possible within the terms of the statute, FDA has a duty to return the problem to Congress for reconsideration. Apparently FDA believes that its suggested procedures, implemented by administrative "interpretation," are within the terms of the statute, and will make possible a reasonably efficient handling of incidental packaging additives. It is proper to ask, however, if these procedures are parts of a carefully studied plan by the FDA management team, including its counsel, or if they are but a succession of reactions to newly crystallized problems. Has thoughtful consideration been given to the legality and the propriety as a policy matter of imposing these procedures upon the producers and consumers of incidental additives? Has FDA thoroughly weighed the cost to the consumer of insisting on continued administration of an inapplicable statute through engrafted and inconsistent interpretations?

REGULATION BY SPECIFICATION

Initially the suggestions offered by FDA were directed toward easing the burden of laboratory analysis. It was suggested, for example, that if the total amount of a migrating substance present in a packaging material would be safe in food, it would not be necessary to determine the actual amount migrating.[19] Another suggestion was that if a given quantity of a migrating chemical would be safe in the food, the analytical test method would not have to be sensitive to amounts below the safe level. Thus, the Regulation could specify a test method of limited sensitivity and if the test showed zero, use of the substance would be in conformity with the Regulation.[20]

If a packaging product were to be approved because the total amount of a particular migrant present in the product would be safe in food, even if it all migrated, it would be necessary to establish specifications for that particular product so that it would not be manufactured later with a greater amount of the particular migrant in it. If specifications were to be established for one ingredient of an end product, FDA soon came to the view that it might be useful to establish specifications for the entire end product. This would have the advantage of permitting, in some cases, an analytical control test of the end product as a whole, and from the point of view of an ardent regulator, of course, it would have the advantage of placing an entire line of commerce under control instead of only one, small, raw material ingredient.

THE "BLANKET REGULATION"

The specification approach, which is exemplified by the polypropylene and polyethylene regulations,[21] ed

[18] Deputy FDA Commissioner John L. Harvey, *FDCLJ* 15, No. 10: 619–620 (October, 1960).
[19] A. A. Checchi, Assistant to the Deputy FDA Commissioner, *FDCLJ* 14, No. 9; 593 (September, 1959).
[20] Ibid at pp. 593–594.
[21] 21 C.F.R. 121.2501 (Polypropylene) and 21 C.F.R. 121.2510 (Polyethylene).

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to another form of regulation which has come to be called the "Blanket Regulation." Although it has been much discussed, only one Regulation in this form has been issued.[22] In the author's opinion, this form of regulation is outside the bounds of the regulatory plan set up by the Food Additives Amendment and its use creates legal and policy problems which FDA has not adequately considered.

The Blanket Regulation was initially described this way:

> " . . . Our people have been discussing with industry representatives the possibility of some sort of blanket regulation for specific types of packaging materials which will list the various components which themselves may be food additives and which may be used provided the packaging material meets certain extraction tests of an exaggerated character using various solvent materials."[23]

As described, the regulation would be similar to the polypropylene-polyethylene technique, involving an extractability test for the end product. It would differ in that only some of the components would be listed and there would be no overall specification. In due course, however, it was suggested that a "Blanket Regulation" might not necessarily involve specific products and might not necessarily involve an extractability test. Instead, it was suggested, a Blanket Regulation might constitute a listing of all the chemicals involved in all the end products made of one primary material, e.g., paper, paperboard, cellophane, etc. with the chemical components grouped according to whether or not they were Generally Recognized As Safe (GRAS), Covered by a Prior Sanction, Not Reasonably Expected to Transfer, or Covered by an Issued, or To-Be-Issued Regulation.[24]

Such a Blanket Regulation would be an ineffective general description accomplishing nothing positive in and of itself. However, such a Blanket Regulation raises more serious legal and policy questions. For example, it is hard to find the legal authority to list in a Food Additive Regulation purportedly issued under Section 409(c) of the statute, many chemical substances which are admittedly completely exempt from Regulation under Section 409 because they are exempt from the definition of "food additive" contained in Section 201(s).

The same question frequently has been raised with reference to Regulation 21 C.F.R. 121.101 in which, by Regulation, FDA purports to establish those substances which are GRAS.[25] GRAS substances, by definition, are not food additives and are totally exempt from regulation as food additives. It is beyond question that FDA is not the authority designated to determine whether any substance is GRAS. Congress set the standard for determining that question, and any manufacturer is entitled to apply it to his own situation. FDA has a duty to reach a judgment on the GRAS status of any suspect substance, and if FDA determines that substance is not GRAS, FDA should continue its inquiry to see if an unlicensed food additive is involved. However, when FDA finds that a substance in GRAS, in FDA's opinion, then that substance, so far as FDA is then concerned, is exempt from the law and from FDA's control or jurisdiction under the Food Additive Amendment.

FDA's practice of issuing Regulations stating which substances are GRAS encourages the impression that no substance is GRAS unless FDA, by Regulation, makes it so. Customers become very uninterested in having a supplier tell them that a given migrant is exempt because it is GRAS unless FDA has included that migrant on one of its published lists. The net result in the commercial world in many product areas has been to preclude individual determinations of GRAS status and to give FDA nearly as much control over exempt substances as it has over those which Congress placed under FDA jurisdiction. Fortunately this trend has been somewhat slowed by the establishment of expert panels in some fields to establish GRAS lists without reference to FDA's views.[26] It would be reassuring if FDA would re-examine, as a policy matter, the entire concept of issuing Regulations which to the commercial world appear to be Section 409(c) Food Additive Regulations, but which are partially or wholely devoted to exempt substances.

FDA's apparent intention to regulate that which Congress has not subjected to regulation appears again in FDA's expressed desire to write Regulations for entire industries and all their products (e.g., paper and paperboard), under a statute which covers only a few of the raw material ingredients used. There is no

[22] For Cellophane, 21 C.F.R. 121.2507.
[23] Deputy FDA Commissioner Harvey, *FDCLJ* 15, No. 10: 620 (October, 1960).
[24] This suggestion has been made to representatives of the American Paper and Pulp Association, the National Paperboard Association, the Paper Shipping Sack Manufacturers Association, and the Glassine and Greaseproof Manufacturers Association.
[25] " . . . generally recognized, among experts qualified by scientific training and experience to evaluate its safety as having been adequately shown . . . to be safe . . . " See Note 5 supra.
[26] See the description of the Flavoring Extract Manufacturers Association panel and its work, given by Franklin M. DePew, president of the Food Law Institute, *FDCLJ* 16, No. 5: 257 (May, 1961).

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evidence that Congress ever intended the FDA should write Regulations stating how paper, paperboard, cellophane, etc. were to be manufactured. Such a prospect was never considered in all the years of hearings on the Food Additive Amendment and most lawyers would agree the statute would need to be very clear before such a drastic degree of regulation could be authorized. Something akin to the authority to standardize foods, so carefully established by Congress in Sections 401 and 701(e) of the Act would be required before FDA would have the power to standardize food papers, food cellophanes, etc. Yet if the Food Additive Regulation itself is written in terms of vast groups of end products and purports to list all the chemicals which may be present in the end product (including large groups of chemicals *not* covered by the law as well as those which *are* subject to it), the same drastic result is achieved without statutory authority.

Lastly and perhaps most important, under the proposed Blanket Regulation procedure there seems to be no requirement that petitions contain reports of toxicological investigations and a description of a practical means for enforcing the Regulation, that is, of readily determining whether or not the product or products to be controlled comply with the Regulation. As previously indicated, it is difficult if not impossible to apply these provisions to incidental additives but these requirements are not merely administrative requests to be waived at FDA's pleasure. The statute itself requires the Petition for a Food Additive Regulation to contain "a description of practicable methods for determining the quantity of such additive in or on food . . ." and ". . . full reports of investigations made with respect to the safety for use of such additives . . ."[27] A Blanket Regulation issued in a form ignoring these statutory requirements is of very questionable value, for such a Regulation would appear to be voidable at any time for failure to conform to the statute; and—if FDA should seek to cure the Regulation's voidable nature—its proponents might at any time be subjected to demands for the missing scientific data, or have their industry subjected to a form of control they never anticipated.

FACTORY INSPECTION: THE "FOOD-ADDITIVES-ARE-FOOD" THEORY

There may be an explanation for the absence of a control method from some of the FDA descriptions of the Blanket Regulation. FDA believes it has, or should have, very comprehensive factory inspection powers. The Polypropylene Regulation seems to assume that there will be inspections by FDA of plants producing this material. Factory inspection may be the implied method for assuring compliance with other Blanket Regulations now contemplated.[28]

Here again serious legal questions arise. The statute contains a power to inspect. Section 704(a) of the Act[29] states in part that for purposes of enforcement, duly designated employees are authorized:

"(1) To enter, at reasonable times, any factory, warehouse,

[27] Sections 409(b) (2) (D) and (E) of the Act, 21 U.S.C. 384(b) (2) (D) and (E).

[28] "Plant inspection can augment laboratory testing to insure compliance with such requirements. It is on this philosophical footing that we have constructed our regulations for polypropylene and the nylons, and are considering those for can enamels and similar substances." L. M. Beacham, FDA Division of Food, *FDCLJ* 16, No. 5: 264 (May, 1961).

[29] 21 U.S.C. 374(a).

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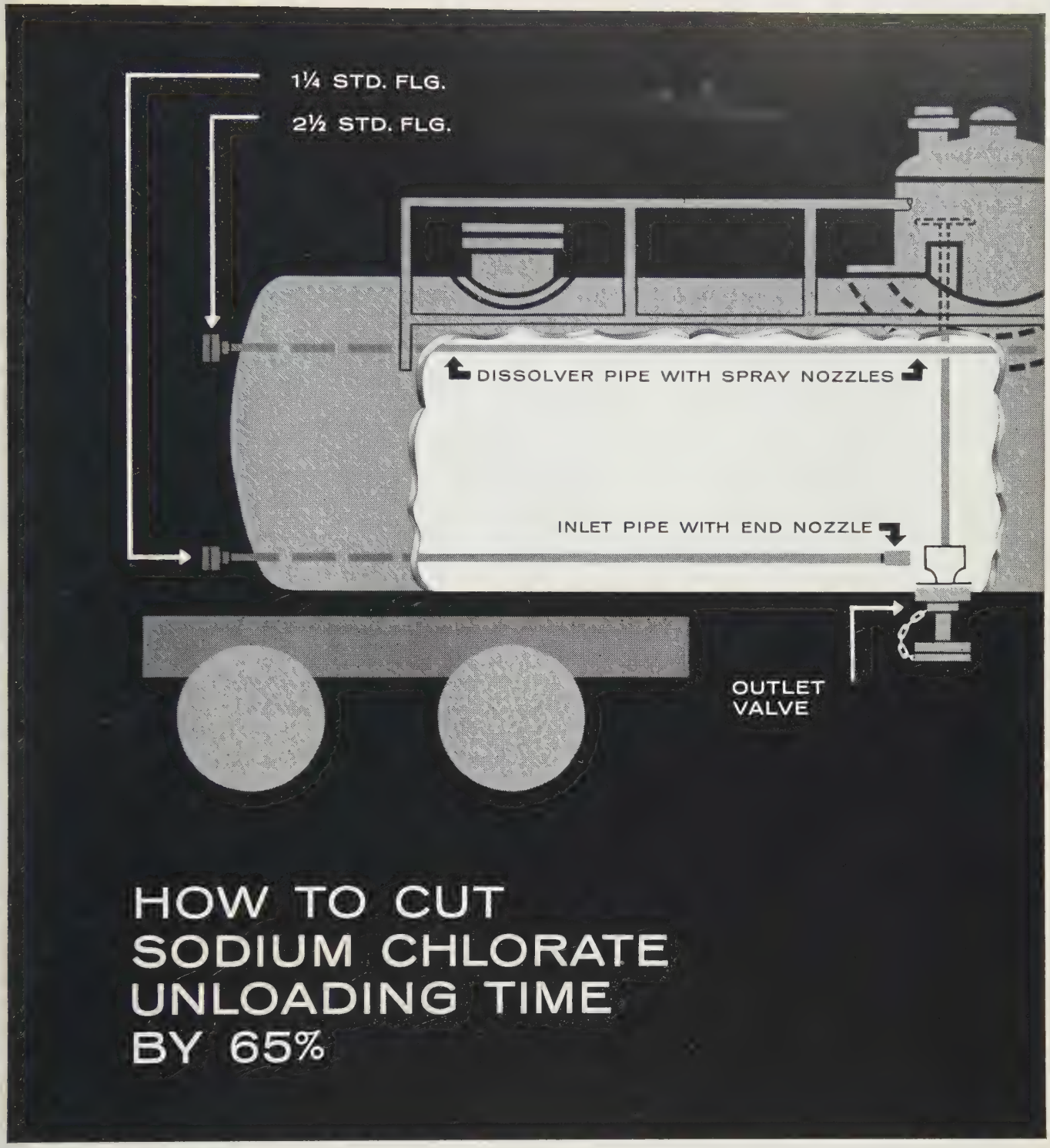

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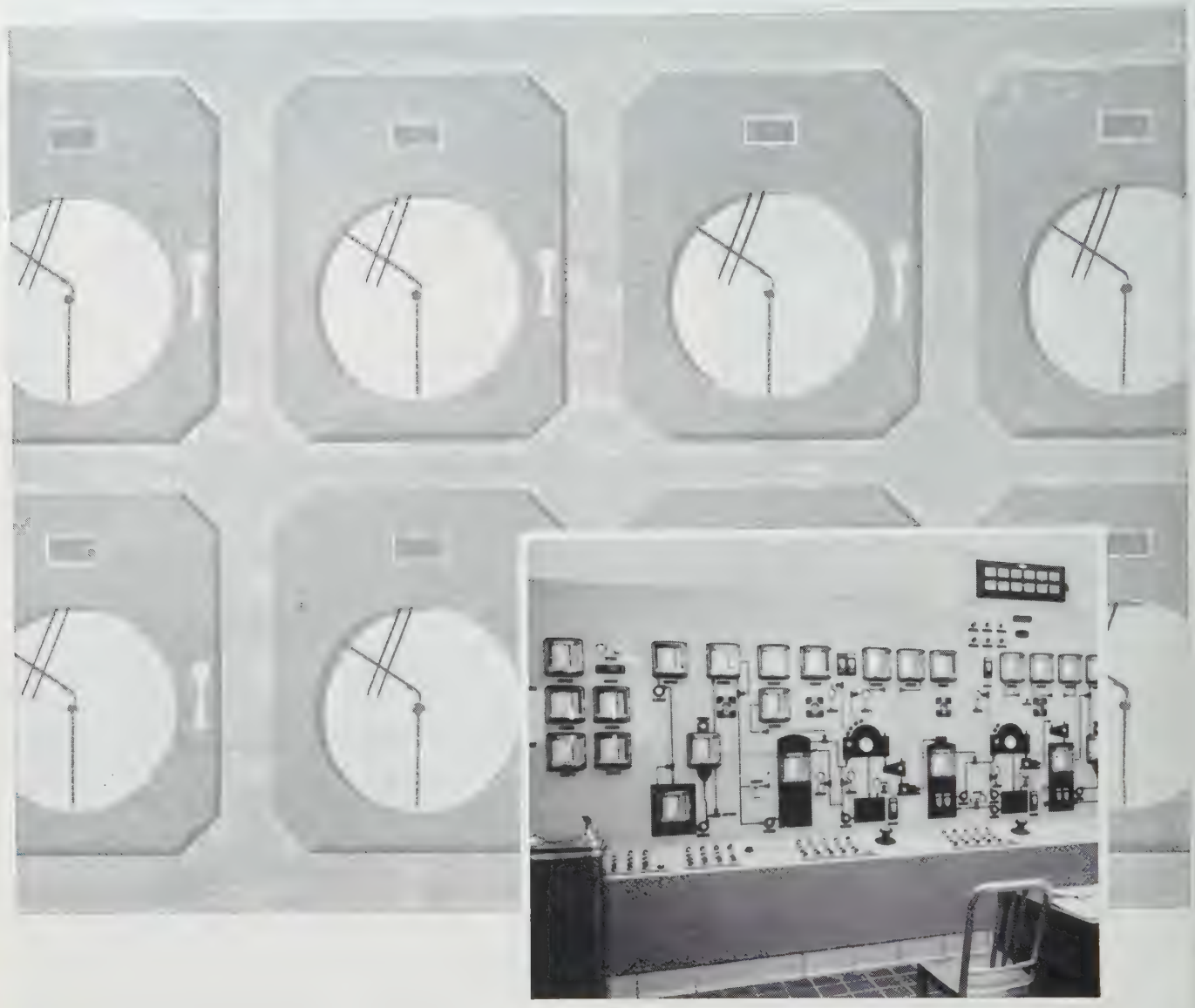

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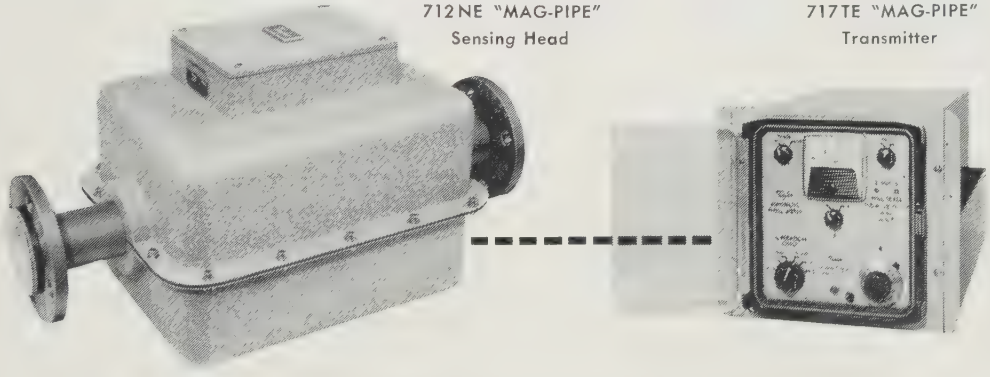

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or establishment in which *food*, drugs, devices or cosmetics are manufactured, processed, packed, or held, for introduction into interstate commerce or are held after such introduction . . . and

"(2) To inspect . . . such factory . . . and all pertinent equipment, finished and unfinished materials, containers and labeling therein." (Emphasis supplied.)

So far as packaging additives are concerned, the key word in the above quotation is "food." There is no explicit authority to inspect factories, warehouses, etc. where food *additives* are manufactured, etc., nor to inspect such establishments where food *containers* are manufactured, etc. Congress never considered, let alone authorized, FDA inspection of industrial chemical factories, packaging plants and other nonfood establishments. When the Food Additive Amendment was passed, no change was made in the authority granted by Section 704(a).

FDA claims to find authority for such inspections in the theory that a "food additive" is a "food" and these two terms are interchangeable throughout the statute. This equation of words arises from the definition of food in Section 201(f) of the Act, which includes "articles used for components" of food, and the definition of food additive in Section 201(s) which refers to substances which may reasonably be expected to become components of food.

FDA has used this same argument as one support for its claimed power to seize unlicensed food additves before they are ever used with food. (The statute authorizes the seizure of adulterated *food*, for instance where a food is adulterated because it bears or contains as unlicensed food additive,[30] but it does not authorize seizures of unlicensed food *additives* capable of some day causing a food to be adulterated.) The theory in this application has been well analyzed—and rejected—by at least one well qualified writer.[31]

In answer to questions about the statutory authority for factory inspection and seizing empty containers (which might be unlicensed food additives) FDA's General Counsel stated the FDA position as follows:

"As far as I'm concerned, it is true that 'food additives' were not specifically made 'food' by definition when the Food Additives Amendment was passed. The Congress' explanation for that was. 'It was unnecessary to do so.' . . . If any container has a poisonous[32] substance in it that is reasonably expected to migrate to food and the container is being shipped to, or is in the possession of, a food processor, we would not hesitate to attempt to take regulatory action to prevent its use before food was packaged in it, thereby rendering the food adulterated. We think the law is ample on that point.[33]

"The question of inspecting these places[34] has not come up. As you know, our inspection authority is supported by a criminal statute and of course is likely to be strictly construed. I must say, however, that if anyone will look at the polypropylene food additive regulation he'll see that it is established in terms of what was used in preparing that substance.

"As a practical matter, we're not going to be able to issue regulations for some packaging material without doing it on the basis of specifications for the packaging material. In order to make those regulations effective there will have to be an adequate inspection power. I don't anticipate any difficulty on this nor am I here suggesting that when we bring our first criminal case to get a reliable interpretation of the factory inspection authority, we go into some can company to start. The statutory basis for authority to inspect would have to be that the food additive is a food because it is intended for use which results in its becoming a component of food . . ."[35]

Packaging additives are not "used for components of food" even though they may "become components of food," and no amount of semantical acrobatics will change that fact. Also, there is no evidence, as has been said, that Congress intended the word "food" in the factory inspection section of the Act to authorize inspections of metal, paper, and plastics packaging plants. In addition to these points, we should note another problem with the "food-additives-are-food" theory: the application of the food labeling provisions of the Act to all food additives. If FDA accepts this theory—and the gains in terms of expanded factory inspections and seizure powers suggest that FDA will push the theory—then why does FDA do nothing about all the "misbranded foods" (i.e., food additives) being shipped every day?

The answer is that FDA recognizes that food additives don't need to be labeled as foods, in many cases could not be so labeled, and that Congress never said they should be so labeled. FDA has said: "We do not believe that an empty can[36] requires labeling as a food."[37] Then, in the same breath, FDA says: "We believe that can manufacturing plants are subject to inspection to detect possible violations of the food-additives amendment."[38] From this it appears that while FDA may want some of the benefits of its "food-additives are-food" theory, it will not apply that theory consistently across the board. If Congress intended that all uses of the word "food" in the statute should include "food additives," FDA would have no such choice. Perhaps even FDA has some reservations about the validity of this theory.

"A SHORT-CUT PROCEDURE"

The FDA originally wanted to hold all additive clearance techniques closely in line with the statute:

" . . . we have been asked if we do not recognize that we should take the position that the incidental additive from packaging materials be classed as '*de minimis*' or of 'pharmacological insignificance.' While these are nice sounding terms, the fact remains that we just are not in a position to solve our food-additives problems on the basis of semantics.

"When you come right down to fundamentals, the proponents of this idea are really asking that we deal with these incidental additives on a basis clearly different from that contemplated by the law. They are saying that we should, in effect, set tolerances for incidental additives by a sort of short-cut procedure—just because the additives are present in quite small quantities."[39]

In lieu of solving the problem with "nice sounding terms," FDA now suggests we solve it with "the Blanket Regulation." This is a solution "clearly different from that contemplated by the law" for it amounts to a proposal (1) to issue Regulations covering chemicals which are totally exempt from control under

[30] Section 304(a) of the Act, 21 U.S.C. 334 (a) reads: "Any article of food . . . that is adulterated . . . shall be liable to be proceeded against . . . on libel of information and condemned . . . " Section 402(a) (2) (C), 21 U.S.C. 342(a) (2) (C) reads: "A food shall be deemed to be adulterated . . . (C) if it is, or it bears or contains, any food additive which is unsafe within the meaning of Section 409 . . . " Section 409 equates "unsafe" to unlicensed, i.e. a use not in conformity with an issued Regulation.

[31] John G. Kuniholm, "Are Empty Containers Food," *FDCLJ* 15, No. 10: 637 (October, 1960).

[32] Since the Food Additives Amendment is completely antithetical to the concept of "poisons," this reference should be to an "unlicensed food additive."

[33] W. W. Goodrich, *FDCLJ* Vol. 16, No. 1: 51 (January, 1961).

[34] Plants producing food-packaging materials.

[35] Op. Cit. Note 33 at p. 57.

[36] In the context, this reference is to an empty can containing a food additive.

[37] "FDA Answers to Questions," *FDCLJ* 15, No. 4: 232 (April, 1960).

[38] Ibid.

[39] Assistant Commissioner Kirk, *FDCLJ* 15, No. 4: 264–265 (April, 1960).

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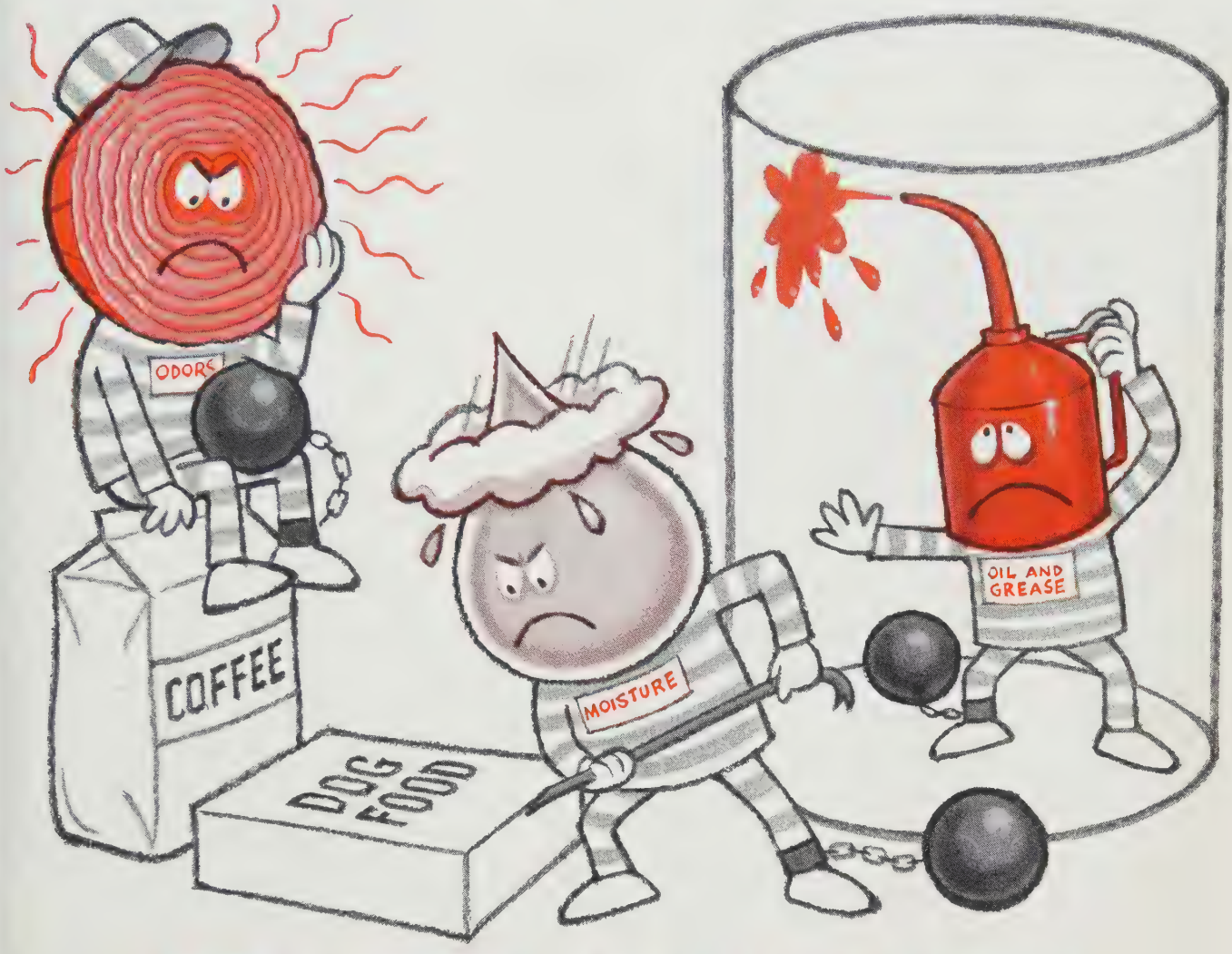

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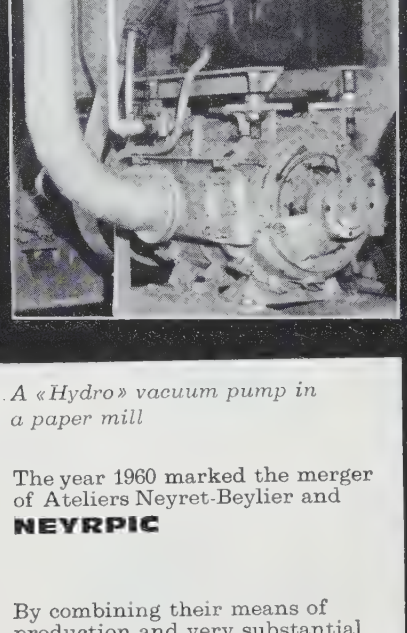

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the enabling statute; (2) to issue Regulations controlling entire industries and the manufacture of their end products under a statute clearly written to deal with only certain chemical ingredients of such end products; and (3) to institute factory inspections of nonfood plants (and seizures of nonfood items) on the basis of a highly questionable statutory construction, inconsistently applied.

This sort of "administrative legislation" does no credit to FDA. Congress always has provided regulatory power where FDA could demonstrate the need. FDA is not overstaffed or short of useful work to do. Thus it is hard to understand why FDA should be so quick to claim additional power beyond that granted by Congress. The Blanket Regulation, inspection of nonfood factories, and seizures of nonfood items may be only reactions to what FDA believes is a regulatory need, but this does not justify proceeding without statutory authority. These activities ought to be closely reviewed by the FDA Commissioner and his policy-making superiors.

A PRUDENT JUDGMENT?

We have been trying to assess the prudence of the judgment that incidental packaging additives should be regulated in the same manner as direct food additives. We have seen the enormous difficulties involved in subjecting packaging additives to the present statutory procedure, and we have seen how this has led the FDA toward forms of Regulation, and methods of enforcement, not sanctioned by the statute. The other results of this judgment cannot be measured with great accuracy but they are beginning to be clear.

First, so far as the author can determine, the huge expenditures and intensive studies of the last three years have not produced any evidence that any old or new packaging material would have been a serious hazard to health if the Food Additives Amendment had not been enacted. Some specifications have been set for new materials, and some limitations have been placed on the usage of components of older materials. However, no major health hazards from packaging materials have been identified, and it is likely that similar specifications and limitations would have been established by the manufacturers involved without governmental fiat—and without the great expense which attended the issuance of formal Regulations.

Second, FDA has not concentrated its efforts on direct additives but has diverted the bulk of its all-too-limited resources to handling incidental additives, and particularly packaging additives. If the direct additive problem is as serious as FDA told Congress it was prior to 1958, it is wasteful (and perhaps even dangerous) to divert effort from that field to incidental additives.

Third, there has been a sharp reduction in research on, and the development of, new products and new methods. The number of research workers and facilities available in industry is limited. If these workers and facilities must be devoted to solving difficult and lengthy analytical problems involving existing products, they cannot be serving the consumer at the same time with the development of better and cheaper new products.[40] When the work they must do on old products

[40] For a discussion of related adverse effects on research in animal drugs and agricultural chemicals, see Hagan, *FDCLJ* 15, No. 2: 118 (February, 1960) and Ringuette, *FDCLJ* 15, No. 5: 334 (May, 1960).

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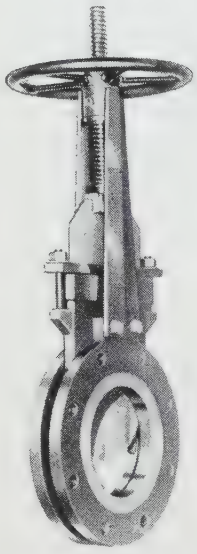

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is enormously expensive, the loss to the consumer in new products is very great.

Fourth, the aggregate cost of the scientific determinations required by the present law for incidental additives is tremendous. That cost is a cost to the consumer. Is the consuming public paying this tremendous cost for a negligible (if any) increase in the safety of what is already by far the world's finest food supply? The foregoing strongly suggests that this is true, and if it is, we are indeed being "improvident in the use of our resources.[41]

CONGRESSIONAL RECONSIDERATION

If, as the author believes, the present regulatory plan is imprudent when applied to incidental packaging additives, Congress should be asked to reconsider it. If some form of control is necessary, an appropriate method of achieving it at a reasonable cost to the consumer can be devised. Or it may be that the evidence will show rather clearly that there are no hazards from packaging additives which justify any form of regulation. In either case, the enormous waste, cost, and the administrative aberrations resulting from the present inapplicable statutory plan will be remedied.

A vigorous effort should be made to enlist FDA's support for Congressional reconsideration, but the effort must come soon, before the ill-considered regulatory policy exemplified by the Blanket Regulation becomes so well established that the FDA will not be willing, as a policy matter, to change its direction. Few governmental agencies have so consistently demonstrated their fair-minded objectiveness over the years, and it is certainly reasonable to expect that FDA's top-level administrators, if they can somehow be caused to take the time to thoughtfully consider the problem, will recognize the need for a special legislative solution and will cooperate in seeking it.

[41] See footnote 3.

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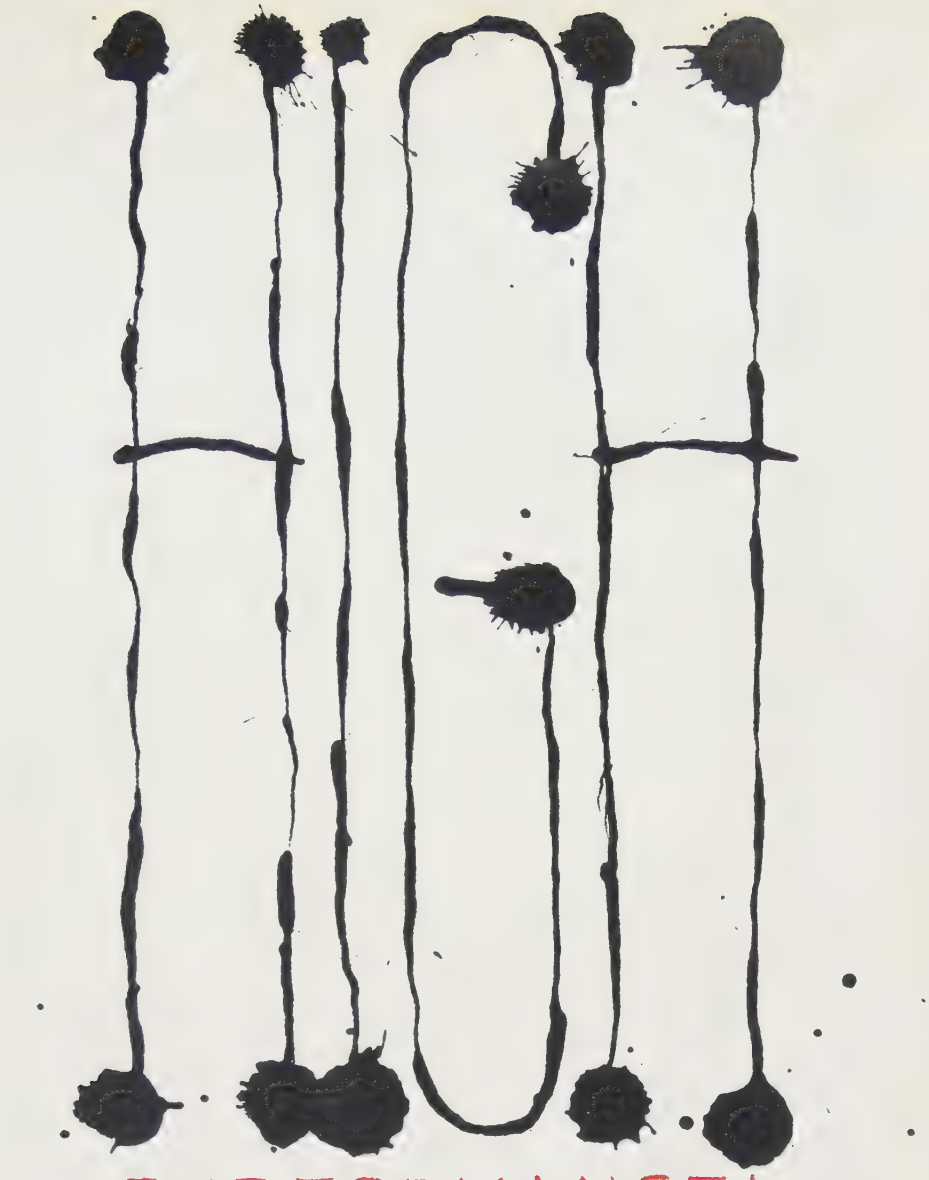

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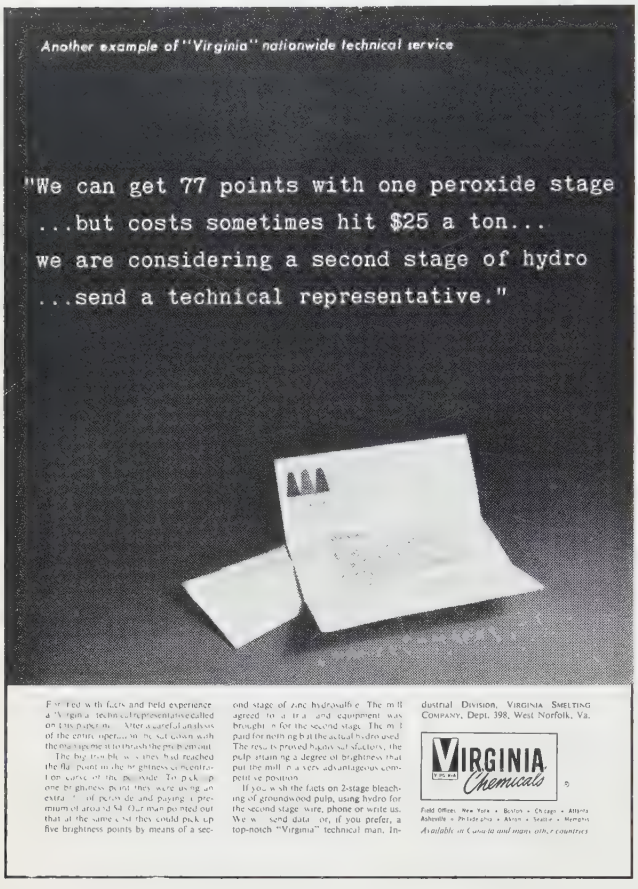
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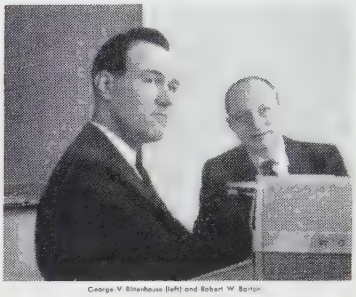

George V. Rittenhouse (left) and Robert W. Barton

Technical representative Bob Barton and salesman George Rittenhouse, both of Virginia Smelting Company, answer some questions regarding the efficiency of bleaching groundwood pulp with one stage of hydro

Q Do zinc and sodium hydrosulfites provide the most economical method of one-stage pulp bleaching?

Mr. Barton—The choice ... between ... points ... with hydro is always the cheapest method. It is ... apply ... are no ... tests involved ... the chemical cost ...

Q Is it difficult to set up a trial run for a mill that might want it?

Mr. Barton—Not usually. Frequently, much of the equipment we need is already available in the mill. We go in with our lab equipment and make tests, checking the *pH* we're going to deal with, and possibly deciding on the maximum temperature possible. We ... all the facts to the mill ... and if they want the trial, we set it up. Often the mill has a pulp storage chest. A few changes and we can use it as a bleaching tank. We'll ... feeders, attach hoses and ... the trial.

Mr. Rittenhouse—Our technical service engineer ... sees there is the set-up ... perfection, and then ... we ... the specialists know everything ... need. ... We're surprised at what we can ... sometimes even in mills ... have been bleaching regularly with hydro. Then ... the personnel ... on a ... to ... of the stock. Of ... hour ... breaks down for ... when it is ... and then ... and up will ... to ... short ... ten ... not ... the ... of ... proper ... and ... brightness ... response at the end. I ... add that faults ... with ... the ... cause a corrosion problem. ... a ... that does not ... proper ... for ... mixing ... standard ... procedure.

Mr. Barton—A good point, George. This is the chief reason why some mills are under the misapprehension that hydro always causes a corrosion problem. If hydro is handled properly there's no corrosion problem.

Q Isn't a mill trial a pretty extensive service at no charge?

Mr. Rittenhouse—Yes, I suppose so—but I consider it the most effective way of showing a mill the economy and the good results obtainable with hydro. After all, mill trial data offer the most believable evidence of what I say. And production doesn't have to be stopped—we can almost always set up the mill trial without affecting it.

Q What are the advantages to the mill in using a "Virginia" hydro?

Mr. Barton—There are several. One is the consistently uniform quality and purity of our hydro, either the zinc or sodium. It's a very clean, free-flowing powder, made pickle-fume shipped in moisture-free ... is made ... manufacturing methods can ... it.

Mr. Rittenhouse—And of course our technical service goes with it. A mill can always call for assistance and get a Virginia technical man. It's the accepted thing with our customers.

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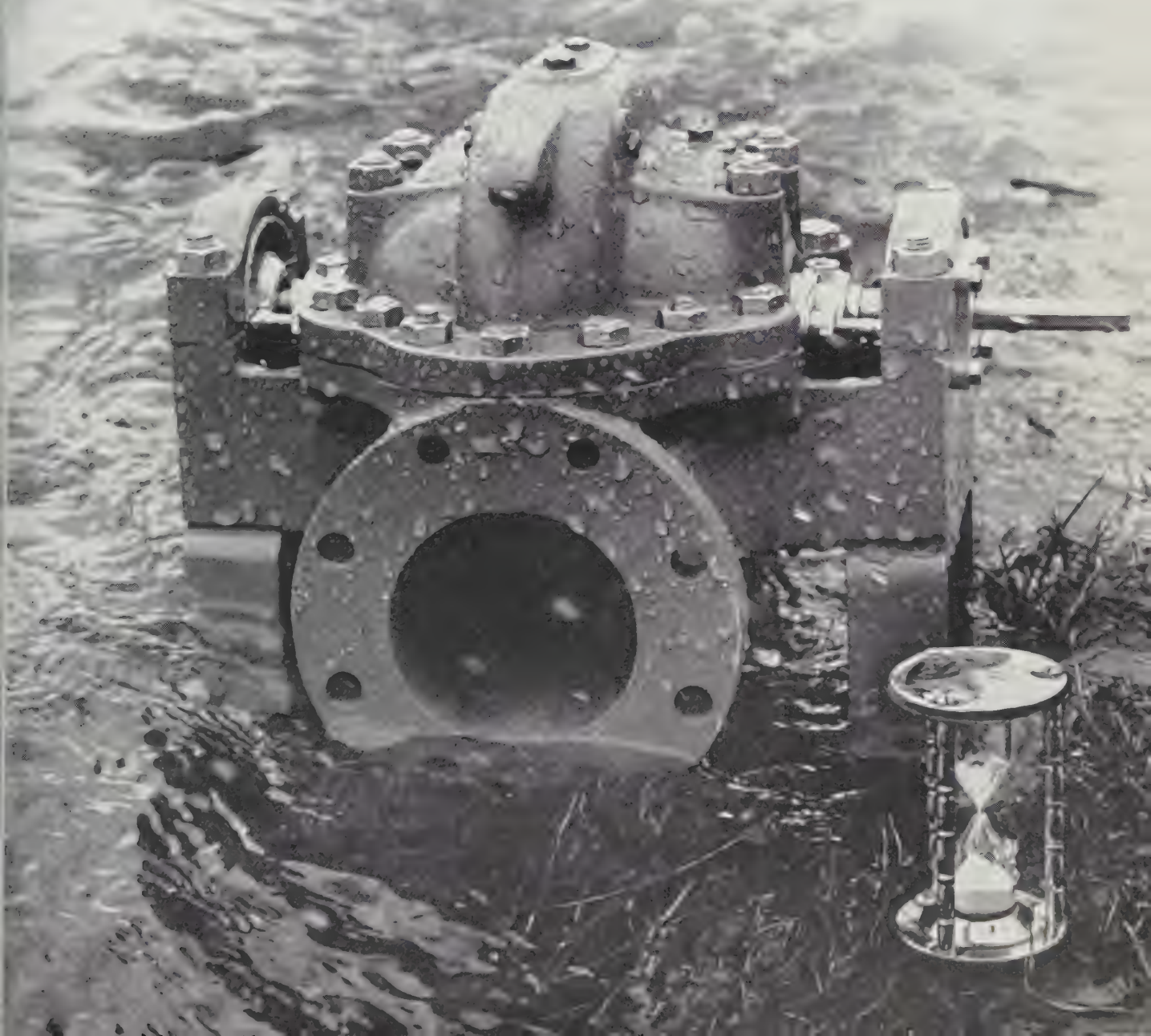

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For the past 26 years the Association has provided funds to enable its committees to undertake research projects. A committee wishing to undertake a research project may request financial aid from the Project Appropriations Committee, which allocates funds. If the committee approves the request, funds are provided by the Association. Upon the discretion of the Project Appropriations Committee additional funds may be granted to continue the project beyond its initial objective. The first grant for a TAPPI research project was made in 1935, when a sum of $150 was authorized for a study of mechanical pulp refiners by fiber classification. Since then more than half a million dollars have been granted for committee projects.

Besides encouraging research as an intellectual pursuit and thus enriching our store of scientific knowledge, many other benefits accrue from such grants: new and immediately applicable technical information is acquired, member interest in the activities of TAPPI technical committees is stimulated, and committees acquire experience in formulating and supervising research programs. In many instances, research work financed by TAPPI has led graduate students to careers in the paper industry. Finally, reports on such research projects have always been a valuable addition to conference programs.

TAPPI policy requires that the results of investigations financed in part or wholly by TAPPI Project Appropriations be made available to the membership of the Association in its publications. Usually these results are presented at TAPPI conferences, followed by publication in *Tappi* magazine. This bibliography was compiled with the help of John L. Parsons, chairman of the Project Appropriations Committee, to enable readers to locate papers resulting from these grants, and to inform them of completed but unpublished research results, as well as projects under way.

In the bibliography, research projects are given chronologically and repeat grants to the same program are grouped together. The committee supervising a research program is shown at the end of the list of references cited under the project appropriation numbers for the program.

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The Association does not have any reprints of published works cited in the bibliography; but copies of *Tappi* containing these works may be purchased from the Association. For recent papers the authors themselves may be able to provide reprints.

BIBLIOGRAPHY

Project Appropriation No.

1 Bray, P. D., Thompson, R. A., and Shyr, Jiun-The fractionation of mechanical pulps and its relation to refining.
Tech. Assoc. Papers 20: 167–68 (June, 1937); *Paper Trade J.* 104, No. 5: 33–34 (Feb. 4, 1937).
Mechanical Pulping Committee

2, 9 Lewis, H. F. Study of existence and nature of pulp membranes.
Tech. Assoc. Papers 19: 24–25 (June, 1936).

Brauns, F. E. and Lewis, H. F. Studies on fiber structure: Physical and chemical characteristics of skin substance or cementing material.
Tech. Assoc. Papers 20: 368B–70B (June, 1937); *Paper Trade J.* 105, No. 10: 35–37 (Sept. 2, 1937).

Lewis, H. F. Existence and nature of fiber membranes: Progress report.
Tech. Assoc. Papers 21: 490 (June, 1938); *Paper Trade J.* 107, No. 16: 36 (Oct. 20, 1938).

Lewis, H. F., Brauns, F. E., and Buchanan, M. A. Existence and nature of fiber membranes.
Tech. Assoc. Papers 22: 475–77 (June, 1939); *Paper Trade J.* 110, No. 5: 36–38 (Feb. 1, 1940).
Fundamental Research Committee

4, 8, 10, 12, 17 Van Beckum, W. G. and Ritter, G. J. Chemical composition of wood.
Paper Trade J. 104, No. 19: 49–50 (May 13, 1937).

Van Beckum, W. G. and Ritter, G. J. Progress report on the chemical properties of holocellulose.
Tech. Assoc. Papers 21: 334 (June, 1938).

Van Beckum, W. G. and Ritter, G. J. Composition of holocellulose, hydrolyzed holocellulose, and Cross and Bevan cellulose.
Tech. Assoc. Papers 22: 619–22 (June, 1939); *Paper Trade J.* 108, No. 7: 27–29 (Feb. 16, 1939), 109, TAPPI Section 299–301 (Nov. 30, 1939).

Hajny, G. J. and Ritter, G. J. Conversion of holocellulose to pulp suitable for high-grade pulp.
Tech. Assoc. Papers 24: 630–33 (June 1941); *Paper Trade J.* 111, No. 22: 131–34 (Nov. 28, 1940).

Hajny, G. J. and Ritter, G. J. Holocellulose research in cooperation with the Technical Association of the Pulp and Paper Industry.
Tech. Assoc. Papers 25: 595–99 (June, 1942); *Paper Trade J.* 113, No. 13: 83–87 (Sept. 25, 1941); *Paper Ind.* 23, No. 11: 1137, 1139, 1141, 1143 (Feb., 1942).
Fibrous Raw Materials Testing and Fundamental Research Committees

5 Calkin, J. Microscopy of paper. TAPPI Special Bibliography Series No. 1, 1937, 47 pages.
Microscopy Committee

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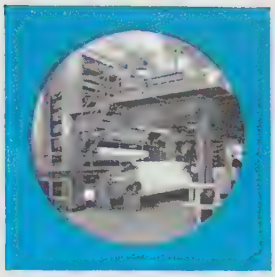

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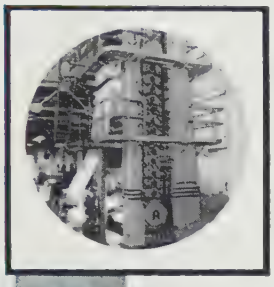

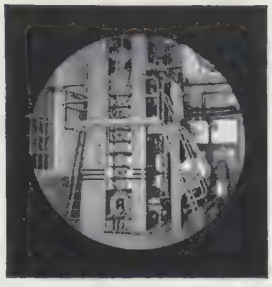

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6 Jahn, E. C. Degree of gelatinization of cellulose and wood.
Tech. Assoc. Papers 21: 350–52 (June, 1938); *Paper Trade J.* 107, No. 9: 38–40 (Sept. 1, 1938).

Sedoff, A., Holmberg, C. V., and Jahn, E. C. Water retention of beaten and gelatinized pulp.
Tech. Assoc. Papers 22: 281–88 (June, 1939); *Paper Trade J.* 109, No. 26: 42–49 (Dec. 28, 1939); *Paper Ind.* 21: 62, 74 (Apr., 1939).
FUNDAMENTAL RESEARCH COMMITTEE

7 Connor, C. M. Effect of alkalinity and impurities on the stability of calcium hypochlorite.
Tech. Assoc. Papers 21: 204–06 (June, 1938); *Paper Trade J.* 107, No. 10: 40–42 (Sept. 8, 1938).
NONFIBROUS MATERIALS COMMITTEE

8 See 4.

9 See 2.

10 See 4.

11, 14, 18 Jahn, E. C. and Holmberg, C. V. Relation of lignin content to the strength of paper and boards: I. Literature survey and progress report.
Tech. Assoc. Papers 23: 626–31 (June, 1940); *Paper Trade J.* 109, No. 13: 30–35 (Sept. 28, 1939); *Paper Ind.* 21: 778, 780 (Oct., 1939).

Holmberg, C. V. and Jahn, E. C. II. Preparation of Pulps of different lignin content by chlorine dioxide.
Tech. Assoc. Papers 23: 128–31 (June, 1940); *Paper Trade J.* 111, No. 1: 33–36 (July 4, 1940); *Paper Ind.* 22: 959–60, 962 (Dec., 1940).

Jahn, E. C. and Holmberg, C. V. III. Pulp sheets and boards prepared from groundwood and delignified by chlorine dioxide, sulfate and soda processes.
Tech. Assoc. Papers 25: 219–24 (June, 1942); *Paper Trade J.* 114, No. 17: 33–38 (Apr. 23, 1942).
FUNDAMENTAL RESEARCH COMMITTEE

12 See 4.

13 Lee, J. A. Survey of materials of construction used in kraft mills and the bleach plant: An investigation of piping in the pulp and paper industry.
Tech. Assoc. Papers 23: 128–31 (June, 1940); *Paper Trade J.* 111: TAPPI Section 1–4 (July 4, 1940).
MATERIALS OF CONSTRUCTION COMMITTEE

14 See 11.

15 Bloom, P. and Jahn, E. C. Determination of cellulose in wood by the use of ethanolamine.
Tech. Assoc. Papers 24: 127–30 (June, 1941).

Bloom, P., Jahn, E. C., and Wise, L. E. Monoethanolamine extraction of wood for the determination of cellulose.
Tech. Assoc. Papers 25: 578–85 (June, 1942); *Paper Trade J.* 115, No. 10: 33–40 (Sept. 3, 1942).
FIBROUS MATERIALS TESTING COMMITTEE

16 Graff, J. H. and Nihlen, E. K. A chart for the estimation of dirt in pulp and paper.
Tech. Assoc. Papers 25: 331–32 (June, 1942); *Paper Trade J.* 114, No. 21: 61–62 (May 21, 1942).
PULP TESTING AND PAPER TESTING COMMITTEES

17 See 4.

18 See 11.

19 Grangaard, D. H., Gladding, E. K., and Purves, C. B. Estimation of the dialdehyde type of oxidation of oxystarches and oxycelluloses.
Tech. Assoc. Papers 25: 385–90 (June, 1942).

Project Appropriation No.

Gladding, E. K. and Purves, C. B. Estimation of carbonyl groups in chromic anhydride oxystarch and oxycellulose by means of hydroxylamine.
Tech. Assoc. Papers 26: 119–24 (June, 1943); *Paper Trade J.* 116, No. 14: 26–31 (Apr. 8, 1943); *Paper Ind.* 25: 306–09 (June, 1943).

Meesok, B. and Purves, C. B. Comparative estimation of carbonyl and carboxyl groups in chromium trioxide and hypochlorous acid oxycellulose and oxyxylans.
Tech. Assoc. Papers 29: 508–15 (June, 1946); *Paper Trade J.* 123, No. 18: 35–42 (Oct. 31, 1946).
FUNDAMENTAL RESEARCH COMMITTEE

20 Waters, V. F. Pipes, pumps, packings and valves in the pulp and paper industry.
Tech. Assoc. Papers 25: 564 (June, 1942); *Paper Trade J.* 113, No. 22: 89–90, 92, 94, 96, 98, 100–104 (Nov. 27, 1941).
MATERIALS OF CONSTRUCTION COMMITTEE

21 Montgomery, A. E. Variation of drying rates of individual driers through the driers section.
Tappi 37, No. 1: 1–13 (Jan., 1954).
DRYING AND VENTILATING COMMITTEE

22 Wells, S. D. Progress in alkaline pulping in 1946.
Tech. Assoc. Papers 30: 591–96 (June, 1947); *Paper Trade J.* 125, No. 18: 42–47 (Oct. 30, 1947).
ALKALINE PULPING COMMITTEE

24, 54 Smith, M. A., Celmer, F., Kraut, J., Witkoski, F. C., and Zuckerman, P. Concerning the Russell formula for gymnosperm lignin.
Tappi 36, No. 8: 342–45 (Aug., 1953).
FUNDAMENTAL RESEARCH COMMITTEE

25, 27, 37, 49, 81, 91, 136, 170 Durst, R. E., Chase, A. J., and Jenness, L. C. An analysis of data on stock flow in pipes.
Tappi 35, No. 12: 529–35 (Dec., 1952).

Durst, R. E. and Jenness, L. C. The flow properties of paper pulp stock: I. The relationship of shear value to pipe friction for bleached sulfite pulp slurries.
Tappi 37, No. 10: 417–22 (Oct., 1954).

Durst, R. E. and Jenness, L. C. The flow properties of paper pulp stock: II. The relationship of shear value to pipe friction for soda, kraft, and groundwood pulp slurries.
Tappi 38, No. 4: 193–98 (Apr., 1955).

Durst, R. E. and Jenness, L. C The flow. properties of paper pulp stock: III. The relationship of shear value to pipe friction for pipes of various diameters.
Tappi 39, No. 5: 277–83 (May, 1956).

Head, V. P. and Durst, R. E. Stock slurry hydraulics.
Tappi 40, No. 12: 931–36 (Dec., 1957).

Durst, R. E. The flow properties of paper pulp stock: IV. The effect of fittings on piping systems carrying pulp slurries.
Tappi 42, No. 9: 713–18 (Sept., 1959).
FLUID MECHANICS AND HYDRAULICS COMMITTEES

26, 45, 70, 129 Browning, B. L., Sell, L. O., and Abel, W. Cellulose solvents for viscosity measurements: The effect of copper and base concentrations in cuprammonium and cupriethylenediamine solutions.
Tappi 37, No. 7: 273–83 (July, 1954).

Browning, B. L. and Sell, O. L. The viscosity of cellulose in cupriethylenediamine solution. Effect of concentration and velocity gradient.
Tappi 39, No. 7: 489–98 (July, 1956).
CHEMICAL METHODS COMMITTEES

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Project Appropriation No.

27 See 25.

28, 47 Project: Thermodynamic properties of ammonium and magnesium bisulfite solutions. Report to be submitted for publication.

ACID PULPING COMMITTEE

29, 41 Standard drainage time of pulp for insulating board. TAPPI Standard T 1002 sm-51.

Hrubesky, C. E. and Perot, J. J., Jr. Evaluation of the SFMC-TAPPI drainage tester.
Tappi 37, No. 10: 425–27 (Oct., 1954).

Haig, D. R. P. A comparison of the TAPPI-SFMC drainage time test procedure with other methods.
Tappi 38, No. 1: 35–38 (Jan., 1955).

Graham, W. H. Evaluation of the TAPPI-SFMC drainage time tester for mill control of insulating board pulps.
Tappi 38, No. 4: 252–55 (Apr., 1955).

Reichman, E. J. Structural fiber—A pulp with its own problems.
Tappi 38, No. 7: 119A–22A (July, 1955).

Jenkins, E. M. Present status of the TAPPI-SFMC drainage time tester for insulating board pulps.
Tappi 40, No. 10: 157A–60A (Oct., 1957).

Coggan, W. C. Unexpected variables affecting the TAPPI-SFMC drainage time tester of Suggested Method T 1002 sm-51.
Tappi 43, No. 8: 723–25 (Aug., 1960).

STRUCTURAL FIBROUS MATERIALS COMMITTEES

30 Rollinson, R. R. and Wise, L. E. A chemical study of the skinlike substance of sulfite pulps.
Tappi 35, No. 11: 139A–40A (Nov., 1952).

FUNDAMENTAL RESEARCH COMMITTEE

Beamesderfer, J. W., Thode, E. F., Chase, A. J. and Hubbard, R. E., Jr. Dye adsorption on wood pulp: I. Adsorption of direct dye on unbeaten sulfite pulp.
Tappi 35, No. 8: 374–379 (Aug., 1952.)

31, 53 Thode, E. F., Beamsderfer, J. W., and Chase, A. J. Dye adsorption on wood pulp: II. Relation of dye adsorption to physical properties of beaten sulfite pulp.
Tappi 35, No. 8: 379–84 (Aug., 1952).

Thode, E. F., Chase, A. J., Majmudar, S. S., and MacKernon, D. R. Dye adsorption on wood pulp: III. Effect of pulp processing on specific adsorption.
Tappi 36, No. 11: 498–504 (Nov., 1953).

Thode, E. F., Chase, A. J., and Hu, Yee. Dye adsorption on wood pulp: IV. Note on effect of drying of pulp on specific dye adsorption.
Tappi 38, No. 2: 88–89 (Feb., 1955).

FUNDAMENTAL RESEARCH COMMITTEE

32 Lewis, W. C. Effects of speed of test on bending strength of insulation fiberboard.
Tappi 38, No. 2: 65–68 (Feb., 1955).

STRUCTURAL FIBROUS MATERIALS COMMITTEE

33 Neisel, R. H. Racking strength and lateral nail resistance of fiberboard sheathing.
Tappi 41, No. 12: 735–37 (Dec., 1958).

STRUCTURAL FIBROUS MATERIALS COMMITTEE

35, 108 Forming structural-type boards for physical tests of pulp. TAPPI Method T 1001 sm-51.

STRUCTURAL FIBROUS MATERIALS COMMITTEE

36 Peoples, R. S. and Ericson, G. L. Corrosion of alkaline pulping digesters: Analysis of corrosion questionnaires.
Tappi 35, No. 9: 402–09 (Sept., 1952).

CHEMICAL ENGINEERING COMMITTEE

37 See 25.

Project Appropriation No.

40, 83 Project: Development of standard methods of analysis of mill waste suspended solids: Determination of Buchner vs. Gooch methods. Results not published.

WATER COMMITTEE

41 See 29.

42 Project: The purchase, alteration and evaluation of a Baumgartner sphere for measurement of the light reflection from insulating board finishes and coatings. Results not published.

STRUCTURAL FIBROUS MATERIALS COMMITTEE

43 Kukachka, B. F. Identification of coniferous woods.
Tappi 42, No. 11: 887–96 (Nov., 1960).

FIBROUS RAW MATERIALS TESTING COMMITTEE

44 Chang, Ying-Pe. Bark structure of North American conifers.
U. S. Dept. Agriculture Technical Bulletin No. 1095 (Dec., 1954).

Anatomy of Common North American pulpwood barks.
TAPPI Monograph No. 14, 1954.

Chang, Ying-Pe and Mitchell, R. L. Chemical composition of common North American pulpwood barks.
Tappi 38, No. 5: 315–20 (May, 1955).

FUNDAMENTAL RESEARCH COMMITTEE

45 See 26.

46 Keller, E. L. and Borlew, P. B. Correlation between chlorine number and lignin content of high-yield kraft pulps.
Tappi 38, No. 6: 379–83 (June, 1955).

ALKALINE PULPING COMMITTEE

47 See 28.

48, 57 Broughton, G. Methods for the determination of cellulose chain-length distribution.
Tappi 37, No. 2: 61–65 (Feb., 1954).

CHEMICAL METHODS COMMITTEE

49 See 25.

50, 67, 71 Ungar, A. and Caywood, T. E. Comparative analysis of digester corrosion measurements.
Tappi 37, No. 5: 177–90 (May, 1954).

Shoumatoff, N. and Teeple, H. O. Comparative analysis of digester corrosion measurements.
Tappi 37, No. 4: 166–71 (Apr., 1954).

Inspection of Digesters. TAPPI Monograph Series No. 12, 1954.

TAPPI Digester Corrosion Subcommittee Summary Report No. 2.
Tappi 38, No. 4: 202–07 (Apr., 1955)

Lientz, J. R., Baisch, S. J., Teeple, H. O., and Shoumatoff, N. Five years investigation of digester corrosion by TAPPI Chemical Engineering Subcommittee.
Tappi 38, No. 6: 373–78 (June, 1955).

CHEMICAL ENGINEERING COMMITTEE

51, 73, 89, 120, 149 Rogers, C., Meyer, J. A., Stannett, V., and Szwarc, M. Studies on the gas and vapor permeability of plastic films and coated papers.

Part I. Determination of the permeability constant.
Tappi 39, No. 11: 737–41 (Nov., 1956).

Part II. Some factors affecting permeability constant.
Tappi 39, No. 11: 741–47 (Nov., 1956).

Part III. The permeation of mixed gases and vapors.
Tappi 40, No. 3: 142–46 (Mar., 1957).

Bhargava, R., Rogers, C. E., Stannett, V , and Szwarc, M. Part IV. Effect of paper substrate.
Tappi 40, No. 7: 564–67 (July, 1957).

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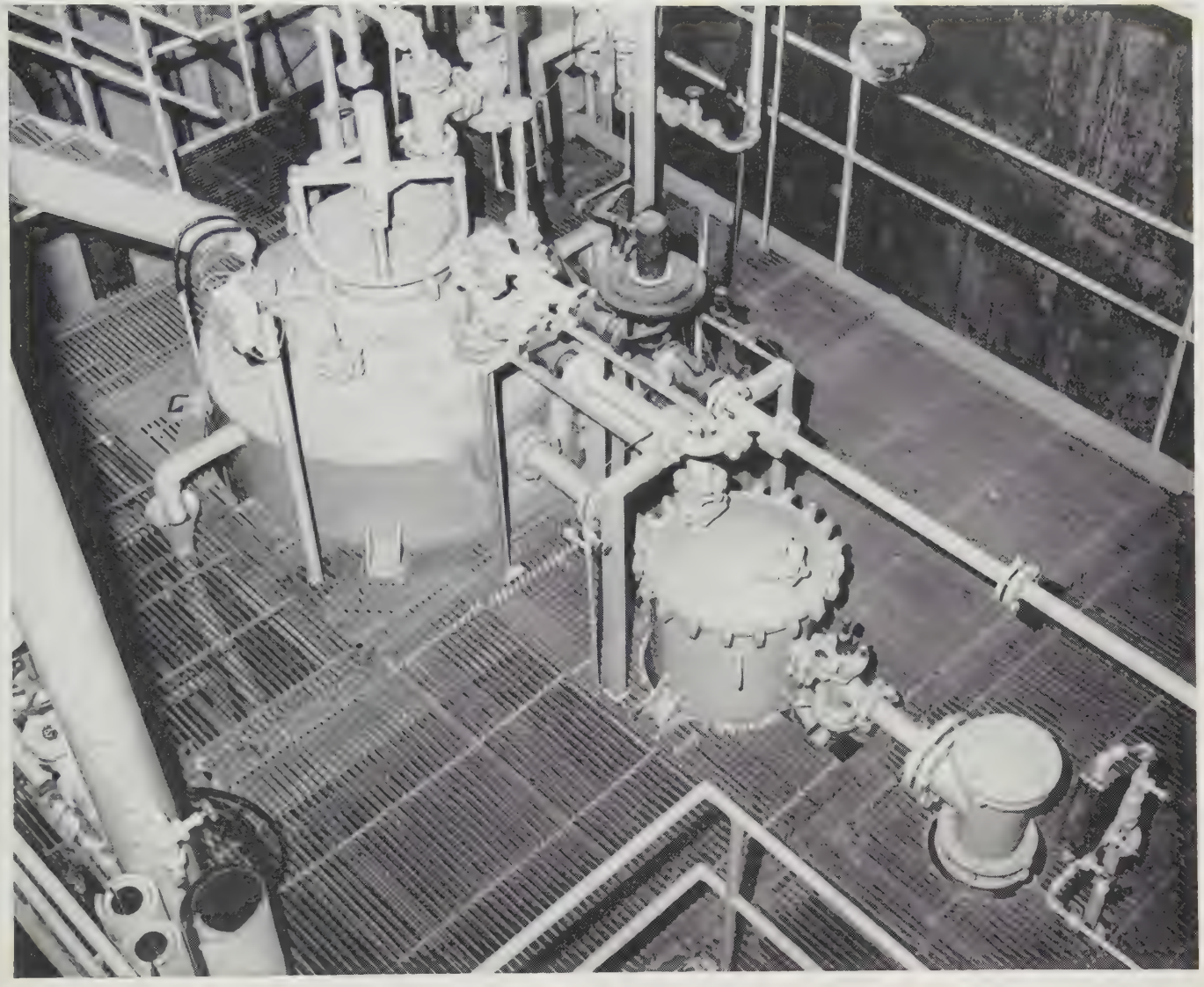

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Project Appropriation No.

Myers, A. W., Rogers, C. E., Stannett, V., and Szwarc, M. Part V. Effect of crystallinity.
Tappi 41, No. 11: 716–20 (Nov., 1958).

Myers, A. W., Meyer, J. A., Rogers, C. E., Stannett, V., and Szwarc, M. Part VI. The permeation of water vapor.
Tappi 44, No. 1: 58–64 (Jan., 1961).

PLASTICS COMMITTEE

53 See 31.

54 See 24.

55 Project: Sulfonation of lignin. Report submitted to Fundamental Research Committee.

FUNDAMENTAL RESEARCH COMMITTTE

56 Stone, J. E. The rheology of cooked wood: I. Introduction and discussion.
Tappi 38, No. 8: 449–51 (Aug., 1955).
II. Effect of temperature.
Tappi 38, No. 8: 452–59 (Aug., 1955).

FUNDAMENTAL RESEARCH COMMITTEE

57 See 48.

58, 74 Broughton, G., Heeks, R. E., and Johannes, C. Methods for cellulose accessibility determination.
Tappi 38, No. 8: 498–502 (Aug., 1955).

CHEMICAL METHODS COMMITTEE

59, 94 Santos, H. A study of the dirt in wood chips.
M.S. thesis, Univ. of Colorado, Boulder, Colo., 1955.

Berkhimer, E. The development of penalty factors for dirt in pulpwood chips.
M.S. thesis, University of Colorado, Boulder, Colo., 1959.

Rowland, T. C. The determination of penalty factors for dirt in pulpwood chips: Development of the procedure.
M.S. thesis, University of Colorado, Boulder, Colo., 1959.

Berkhimer, E., Rowland, T. C., Santos, H. B., and Lauer, B. E. The development of dirt contribution factors for dirt in pulpwood chips.
Tappi 43, No. 12: 989–93 (Dec., 1960).

Irwin, D. and Lauer, B. E. Dirt contribution values for natural dirt in coniferous woods.
Tappi 44, No. 1: 33–35 (Jan., 1961).

FIBROUS MATERIALS TESTING COMMITTEE

60 Mithel, B. B., Webster, G. H., and Rapson, W. H. The action of water on cellulose between 100 and 225°C.
Tappi 40, No. 1: 1–4 (Jan., 1957).

FUNDAMENTAL RESEARCH COMMITTEE

63, 109 Bruce, H. D. and Meniutti, V. P. Small tunnel furnace test for measuring surface flammability.
ASTM Bulletin No. 230: 61–68 (May, 1958).

Bruce, H. D. and Downs, L. E. Surface flammability of various wood-base building materials.
U. S. Dept. of Agriculture Report No. 2140, Forest Products Laboratory, Madison, Wis.
Standard method to be published by ASTM.

STRUCTURAL FIBROUS MATERIALS COMMITTEE

66 Fleming, R., Gaskill, H., and Scott, W. Project: Thermodynamic properties of the systems sulfur dioxide–water-ammonia; sulfur dioxide–water-sodium oxide; sulfur dioxide–water-magnesium oxide; and sulfur dioxide–water-calcium oxide.
Results not published.

ACID PULPING COMMITTEE

67 See 50.

68, 96, 116, 165 Arlt, H. G., Jr., Gross, S. K., and Schuerch, C. A study of the lignin fraction obtained from the alkaline hydrogenation of maplewood.
Tappi 41, No. 2: 64–70 (Feb., 1958).

Bhattacharya, A., Sondheimer, E., and Schuerch, C. Lignin from the alkaline hydrogenation of sugar maplewood: II. The acidic and water soluble fractions.
Tappi 42, No. 6: 446–48 (June, 1959).

Bhattacharya, A. and Schuerch, C. Lignin from the alkaline hydrogenation of sugar maplewood: III. Chromatographic separation of higher-molecular-weight fractions and a structural study.
Tappi 43, No. 10: 840–44 (Oct., 1960).

FUNDAMENTAL RESEARCH COMMITTEE

69 McKee, R. C. and Altmann, F. J. Comparative evaluation of panel, or body-creasing wheel contours.
Tappi 39, No. 7: 503–14 (July, 1956).

CORRUGATED CONTAINER COMMITTEE

70 See 26.

71 See 50.

72, 100, 111, 144, 155, 169, 179 Chase, R. C. A correlation for the Hercules, Hagan, and Brookfield viscometers for a Newtonian fluid.
Tappi 42, No. 2: 97–104 (Feb., 1959).

Shwang, Hsia. The effect of particle size and particle size distribution of clay on rheology of coating colors.
M.S. thesis, University of Maine, Orono, Me., 1958.

Rummel, F. A. The effect of particle size and particle size distribution on the rheology of clay slurries and coating clay colors.
M.S. thesis, University of Maine, Orono, Me., 1959.

Packard, H. N. The characteristics of clay-glucose suspensions as determined by the roll inclined plane technique.
M.S. thesis, University of Maine, Orono, Me., 1960.

COATING COMMITTEE

73 See 51.

74 See 58.

75, 97, 160 Cirillo, G. J. The action of chlorine on glucose alkyl glucosides: I. The quantitative reduction of D-gluconic-δ-lactone to glucose with $NaBH_6$.
Ph.D. thesis, Boston University, Charles River Campus, Boston, Mass., 1960.

Urquiza, M. and Lichtin, N. N. The action of chlorine on glucose and alkyl glucosides: I. The quantitative reduction of D-gluconic-δ-lactone to glucose with $NaBH_4$.
Tappi 44, No. 3: 221–23 (Mar., 1961).

FUNDAMENTAL RESEARCH COMMITTEE

76 Ball, D. H., Jones, J. K. N., Nicholson, W. H., and Painter, T. J. The structure of the hemicelluloses of loblolly pine.
Tappi 39, No. 6: 438–43 (June, 1956).

FUNDAMENTAL RESEARCH COMMITTEE

77, 98, 123, 146, 164, 177 Wolfrom, M. L., Dacons, J. C., and Fields, D. L. The cellodextrins: Preparation and properties.
Tappi 39, No. 11: 803–06 (Nov., 1956).

Wolfrom, M. L. and Fields, D. L. A polymer-homologous series of D-acetates from cellulose.
Tappi 40, No. 5: 335–37 (May, 1957).

Wolfrom, M. L. and Fields, D. L. A polymer-homologous series of oligosaccharide alditols from cellulose.
Tappi 41, No. 5: 204–07 (May, 1958).

Fields, D. L. The cellodextrins; their D-acetates and alditols.
Ph.D. thesis, Ohio State University, Columbus, Ohio, 1958. Final report to be prepared for *Tappi*.

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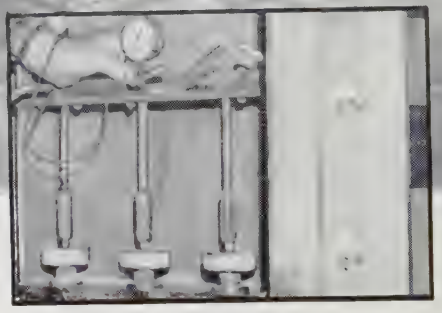

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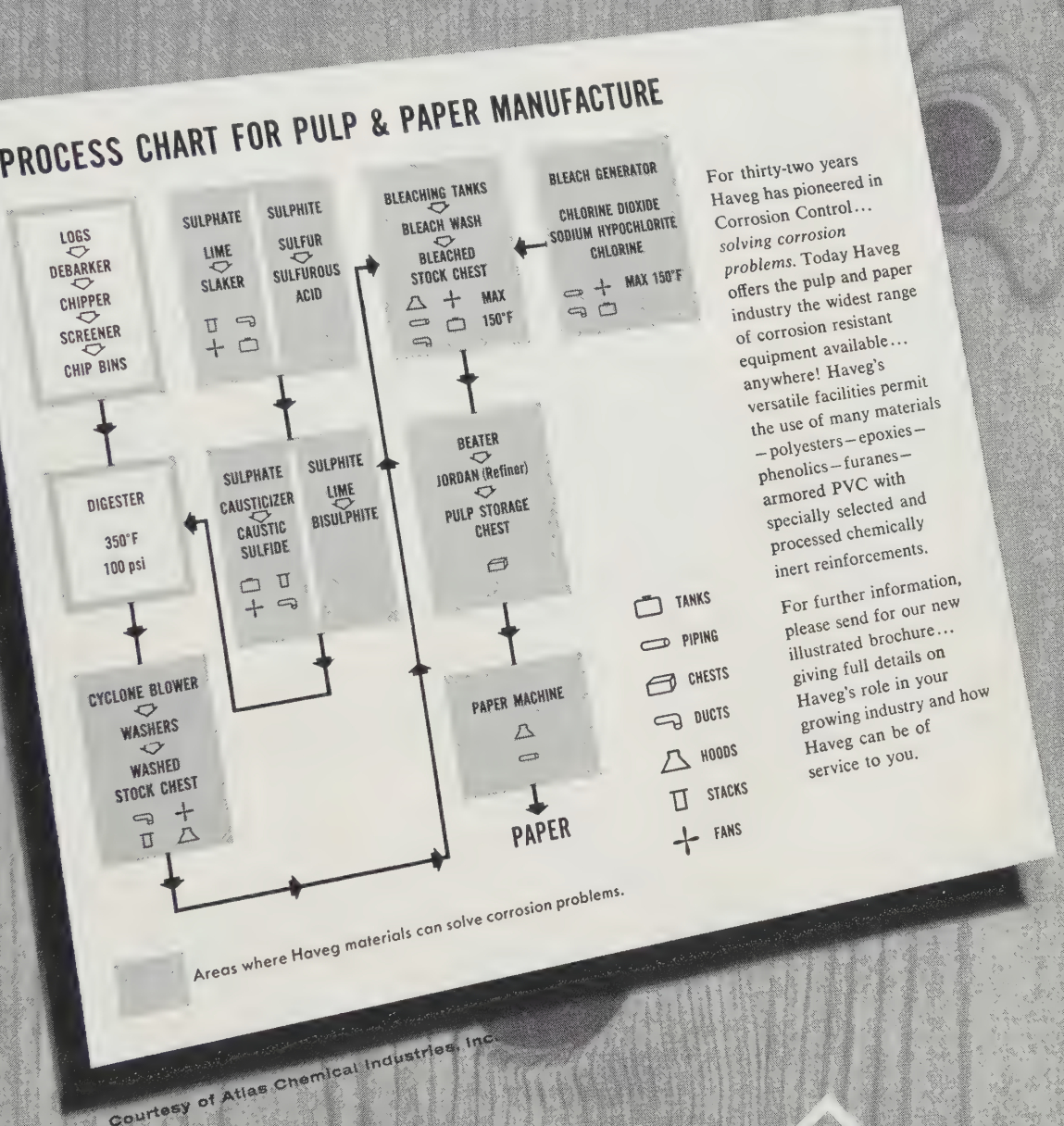

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Project Appropriation No.	
78, 103, 124	Fujita, T. Model Compounds for lignin: The behavior of lignin model compounds with nucleophilic reagents. Ph.D. thesis, Reed College, Portland, Ore., 1956. Loeb, T. Model compounds for lignin. Reactions of apocynol. Ph.D. thesis, Reed College, Portland, Ore., 1957. Final report to be prepared. FUNDAMENTAL RESEARCH COMMITTEE
79, 99, 115	Field, L., Drummond, P. E., and Riggins, P. H., with Jones, E. A. The organic chemistry of sulfur in the kraft process: I. Pine thiolignin as a mixture. *Tappi* 41, No. 12: 721–27 (Dec., 1958). Field, L., and Drummond, P. E., with Jones, E. A. The organic chemistry of sulfur in the kraft process: II. Nature of the sulfur linkages in pine thiolignin. *Tappi* 41, No. 12: 727–33 (Dec., 1958). FUNDAMENTAL RESEARCH COMMITTEE
80	Swanson, J. W. Beater adhesives and fiber bonding—the need for further research. *Tappi* 39, No. 5: 257–70 (May, 1956). FUNDAMENTAL RESEARCH COMMITTEE
81	See 25.
83	See 40.
84	Cundelan, J. V. Power requirements of paper machines. TAPPI Special Report No. 424, Mar. 20, 1958. Cundelan, J. V. Power requirements of paper machines. Final Report—Project No. 358. TAPPI Electrical Engineering Committee. *Tappi* 41, No. 9: 138A–45A (Sept., 1958). ELECTRICAL ENGINEERING COMMITTEE
85, 105, 135	Brandon, C. E. Precision of TAPPI Standard Testing Methods. *Tappi* 40, No. 3: 168A–72A (Mar., 1957). Brandon, C. E. TAPPI reference pulp. *Tappi* 40, No. 12: 178A–80A (Dec., 1957); *Tappi* 41, No. 7: 120A–24A (July, 1958). Brandon, C. E. The precision of the TAPPI standard beater test. *Tappi* 41, No. 9: 129A–32A (Sept., 1958). Bicking, C. A. Precision data available on commonly used TAPPI Standard Methods of test. *Tappi* 42, No. 1: 89A–91A (Jan., 1959). PRECISION COMMITTEE
86	Kesler, R. B. Analysis of alkaline pulping liquors: I. Sulfur compounds in neutral sulfite and kraft white liquors. *Tappi* 40, No. 10: 802–09 (Oct., 1957). Kesler, R. B. and Bakken, J. F. Corrosion of mild steel in alkaline pulping liquors: I. Kraft white liquors. *Tappi* 41, No. 3: 97–102 (Mar., 1958). Kesler, R. B. Corrosion of mild steel in alkaline pulping liquors: II. Neutral sulfite cooking liquor. *Tappi* 41, No. 3: 102–09 (Mar., 1958). Kesler, R. B. Corrosion of mild steel in alkaline pulping liquors: III. Special effect of sulfite ion in kraft white liquor. *Tappi* 43, No. 4: 355–57 (April, 1960). CORROSION COMMITTEE
87	Hunter, R. S. and Lofland, C. A gloss test for waxed paper. *Tappi* 39, No. 12: 833–41 (Dec., 1956). WAX TESTING COMMITTEE
88	Nokihara, E., Tuttle, M. J., Felicetta, V. F., and McCarthy, J. L. Lignin: VIII. Molecular weights of lignin sulfonates during delignification by bisulfite-sulfurous acid solutions. *J. Am. Chem. Soc.* 79, No. 16: 4495–499 (Aug. 20, 1957). Felicetta, V. F. and McCarthy, J. L. Lignin: IX. Molecular weights of lignin sulfonates as influenced by certain acidic conditions. *J. Am. Chem. Soc.* 79, No. 16: 4499–502 (Aug. 20, 1957). ACID PULPING COMMITTEE
89	See 51.
90, 119	Dillingham, E. O., Knuth, D. T., and Weissman, G. E. A manometric method for the rapid practical determination of biochemical oxygen demand. *Tappi* 41, No. 7: 321–33 (July, 1958). MICROBIOLOGICAL COMMITTEE
91	See 25.
92, 140, 156, 181	McNown, J. S. and Spengos, A. A preliminary study of the instability of the stock on the wire of a fourdrinier paper machine. *Tappi* 41, No. 1: 9–11 (Jan., 1958). Yih, C. S. and Spengos, A. C. Free-surface instability. *Tappi* 42, No. 5: 398–403 (May, 1959). Yih, C. S. Instability of a rotating liquid film with a free surface. *Proc. Royal Soc.*, Ser. A, 258, 63–86 (1960). FLUID MECHANICS AND HYDRAULICS COMMITTEES
93, 126, 147, 172	Troutman, W. W. Rheological properties of dilute stock. Interim progress report on Project Appropriation No. 93, MIT, February, 1957. Ippen, A. T., Daily, J. W., and Bugliarello, G. Pertinent factors in flow research on dilute fiber suspensions. *Tappi* 40, No. 6: 478–85 (June, 1957). Daily, J. W. and Bugliarello, G. The characteristics of flow with dilute fiber suspensions. A progress report of work under Project Appropriation No. 93, MIT Hydrodynamics Laboratory, Technical Note No. 2 (Dec., 1957). Institute of Paper Chemistry, Appleton, Wis., Physical properties of fibers used in MIT study under Project Appropriation No. 93. Mimeographed Report, 1958. Daily, J. W. and Tsuchiya, A. Flow of nylon fiber suspensions. A Progress report on a phase of work under Project Appropriation No. 147, MIT Hydrodynamic Laboratory Technical Note No. 4, Feb., 1959. Daily, J. W. and Bugliarello, G. Basic data for for dilute fiber suspensions in uniform flow with shear. *Tappi* 44, No. 7: 497–512 (July, 1961). Bugliarello, G. and Daily, J. W. Rheological models and laminar shear flow of fiber suspensions. *Tappi*, in press. FLUID MECHANICS COMMITTEE
94	See 59.
96	See 68.
97	See 75.
98	See 77.
99	See 79.
100	See 72.
101, 117, 161	Thode, E. F., Swanson, J. W., Kurath, S. F. Mechanism of retention of wet-strength resins: I. Kinetic studies of melamine resin adsorption. *Tappi* 42, No. 3: 170–74 (Mar., 1959). Kurath, S. F., Chu, C. Y., and Swanson, J. W. Mechanism of retention of wet-strength resins: II. Rate studies on the adsorption of urea-formaldehyde resin. *Tappi* 42, No. 3: 175–79 (Mar., 1959).

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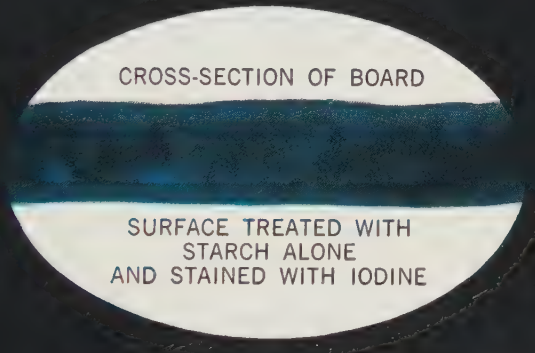

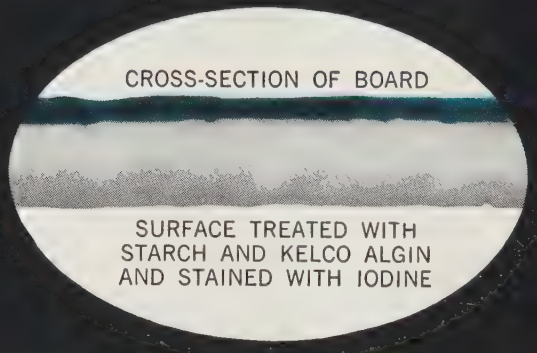

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Becher, J. J., Hoffman, C. R., and Swanson, J. W. Mechanism of retention of wet-strength resins: III. The rate-controlling step in the retention process.
Tappi 44, No. 4: 296–304 (Apr., 1961).
WET STRENGTH AND INTERFIBER BONDING COMMITTEE

102, 118 Jurecic, A., Lindh, T., Church, S. E., and Stannett, V. Studies on the mechanism of wet-strength development: I.
Tappi 41, No. 9: 465–68 (Sept., 1958).
Jurecic, A., Hou, C. M., Sarkanen, K., Donofrio, C. P., and Stannett, V. Studies on the mechanism of wet-strength development: II.
Tappi 43, No. 10: 861–65 (Oct., 1960).
Hazard, S. J., Jr., O'Neil, F. W., and Stannett, V. Studies on the mechanism of wet-strength development: III.
Tappi 44, No. 1: 35–38 (Jan., 1961).
WET STRENGTH AND INTERFIBER BONDING COMMITTEE

103 See 78.

104, 121 McKeough, P. J., Huebner, C. W., and Marshall, W. R., Jr. Wet-bulb hygrometry for the air-water vapor-sulfur dioxide system.
Tappi 44, No. 5: 343–51 (May, 1961).
CHEMICAL ENGINEERING COMMITTEE

105 See 85.

106, 125 Hunter, R. S. Standardization of test for specular gloss of paper at 75°.
TAPPI Standard T 480 m-51.
Tappi 41, No. 8: 385–96 (Aug., 1958).
OPTICAL PROPERTIES COMMITTEE

107 Ziegler, R. D. Procedure for measuring the water vapor permeability of insulation board.
Tappi 40, No. 11: 881–84 (Nov., 1957).
STRUCTURAL FIBROUS MATERIALS COMMITTEE

108 See 35.
109 See 63.
111 See 72.
115 See 79.
116 See 68.
117 See 101.
118 See 102.
119 See 90.
120 See 51.
121 See 104.
123 See 77.
124 See 78.
125 See 106.
126 See 93.
129 See 26.

130, 166 Schaeffer, W. D., Fetsko, J. M., and Zettlemoyer, A. C. Reproducibility studies of pick measurements.
Paper presented at the 44th Annual Meeting of TAPPI, New York, N. Y., Feb., 1959.
Zettlemoyer, A. C., Schaeffer, W. D., Fetsko, J. M., and Grund, G. M. Surface strength evaluation.
Summary report for the year Feb. 1, 1958–Feb. 1, 1959.
Zettlemoyer, A. C., Schaeffer, W. D., Fetsko, J. M., and Grund, C. M. Surface strength evaluation: Round robin pick measurements on offset machine-coated papers.
Progress Report No. 2: Sept. 1, 1958–Feb. 1, 1959, Lehigh University, Bethlehem, Pa.
Moore, C. E. Instrument evaluation II. Pick measurement studies.
Project Report No. 31. National Printing Ink Research Institute, Lehigh University, Bethlehem, Pa.
Progress reports in the form of technical papers were presented by Zettlemoyer, *et al.*, in the Graphic Arts Session at the 45th Annual Meeting of TAPPI, New York, N. Y., Feb., 1960, and by Glassman and Walker in the Graphic Arts Session of the 11th Coating Conference, Chicago, Ill., May, 1960.
GRAPHIC ARTS COMMITTEE

133 Jaskulski, T. M. Experiment to determine the effect of some variables in flat crush testing of corrugated fiberboard. Container Laboratories, Inc., Chicago Div.
Report on Job No. 11835, Mar. 31, 1958. Report No. 11835 in TAPPI file; to be published.
CONTAINER TESTING COMMITTEE

135 See 85.
136 See 25.

139, 167 Institute of Paper Chemistry. Smoothness and compressibility of paper and paperboard as related to printability.
Progress Report No. 1, Project 2060, Sept. 25, 1958.
Institute of Paper Chemistry. Smoothness of paper and paperboard as related to printability.
Progress Report No. 2, Project 2060, Jan. 22, 1960.
PRINTABILITY AND RESEARCH, JOINT ADVISORY COMMITTEE

140 See 92.

143, 171 Lashof, T. W. and Mandel, J. Measurement of the smoothness of paper.
Tappi 43, No. 5: 385–99 (May, 1960). Project nearly completed.
PAPER TESTING COMMITTEE

144 See 72.
146 See 77.
147 See 93.
149 See 51.

153, 176 Murphy, R. A., Kakehi, K., and Sarkanen, K. Studies on the mechanism of chlorine dioxide bleaching.
Tappi 44, No. 7: 465–67 (July, 1961).
Sarkanen, K. J., Kakehi, K., Murphy, R. A., and White, H. Studies on oxidative delignification mechanisms. I. Oxidation of vanillin with chlorine dioxide.
Tappi, in press.
WOOD CHEMISTRY COMMITTEE

154 Preparation of a critical report analyzing information available in pulp and paper brightness recession.
To be included in TAPPI Monograph on Pulp Bleaching.
PULP PURIFICATION COMMITTEE

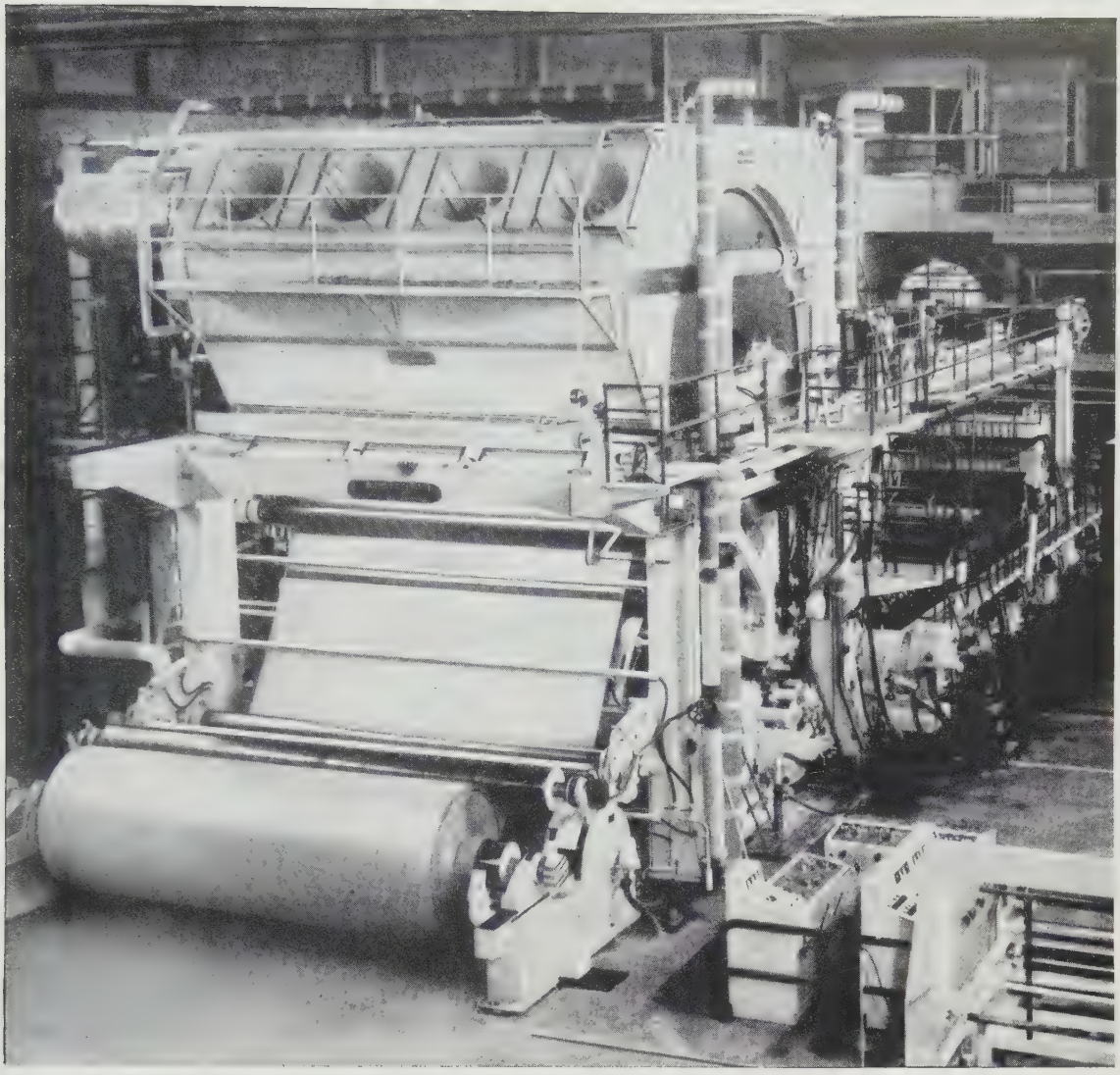
155 See 72.
156 See 92.
160 See 75.
161 See 101.

162 Project: The effect of closed white water systems on groundwood brightness by determination of factors causing discoloration.
Results reported to Mechanical Pulping Committee. To be published.
MECHANICAL PULPING COMMITTEE

163 Project: Sampling plans for the acceptance or rejection of skid lots of paper.
Attributes sampling program-mill survey report. TAPPI Special Report No. 430, Aug., 1961.
STATISTICS COMMITTEE

164 See 77.
166 See 130.
167 See 139.

168, 182 Project: Transfer and penetration of ink receptivity.
Results reported to the Graphic Arts Subcommittee on Ink Receptivity.
GRAPHIC ARTS COMMITTEE



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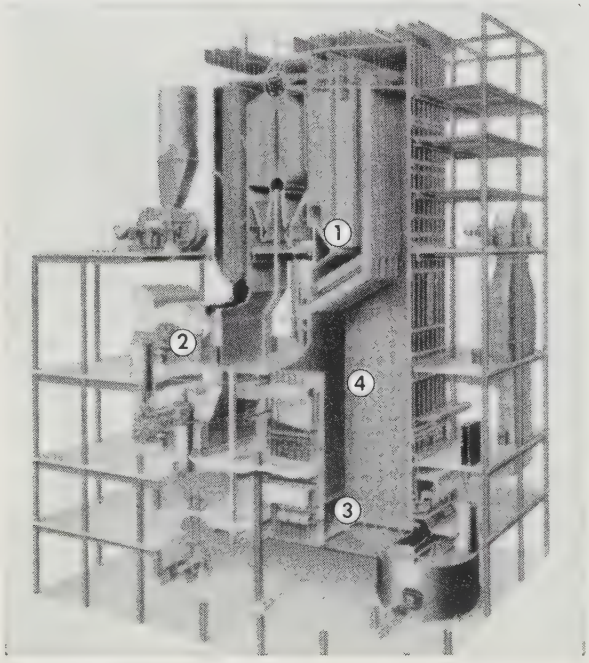

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171	See 143.
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176	See 153.
177	See 77.
179	See 72.
180	Project: The crystallinity of cellulose—a microscopic study. Work in progress. Fundamental Research Committee
181	See 92.
182	See 168.
183	Project: Mechanism of swelling of cellulose in different media. Work in progress. Dissolving Pulp Committee
184	Project: Sulfur dioxide absorption with simultaneous chemical reaction. Work in progress. Chemical Engineering Committee
186	Project: The chemical structure of colored bodies in wood. Work in progress. Wood Chemistry Committee
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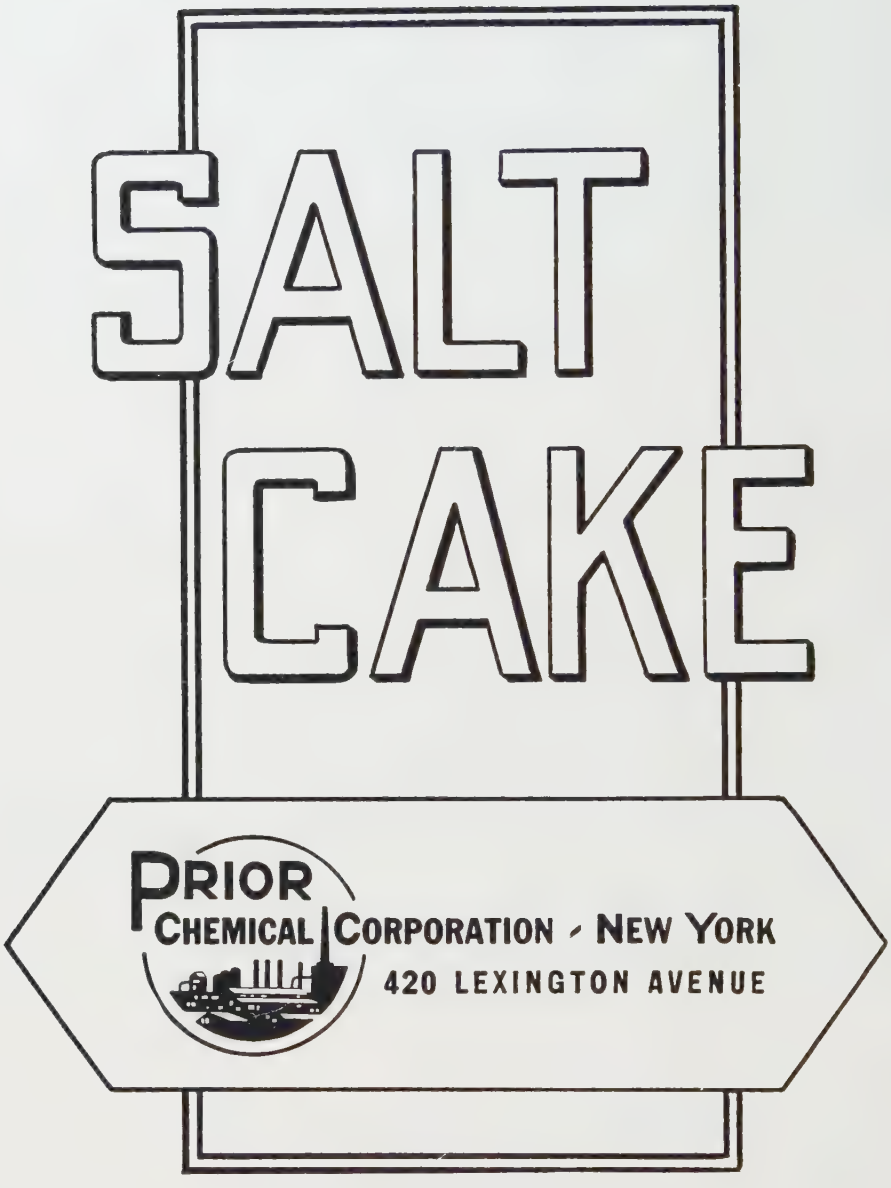
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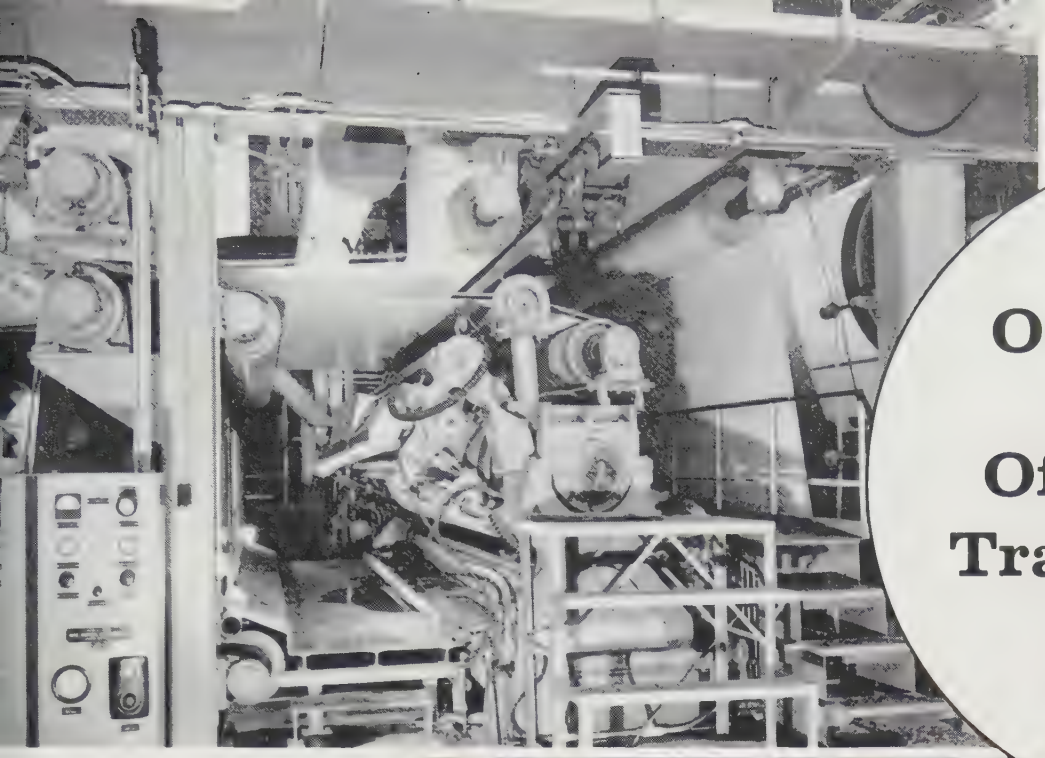

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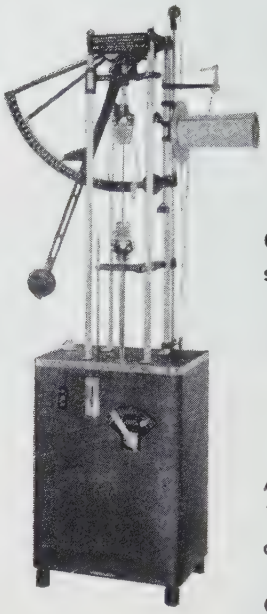

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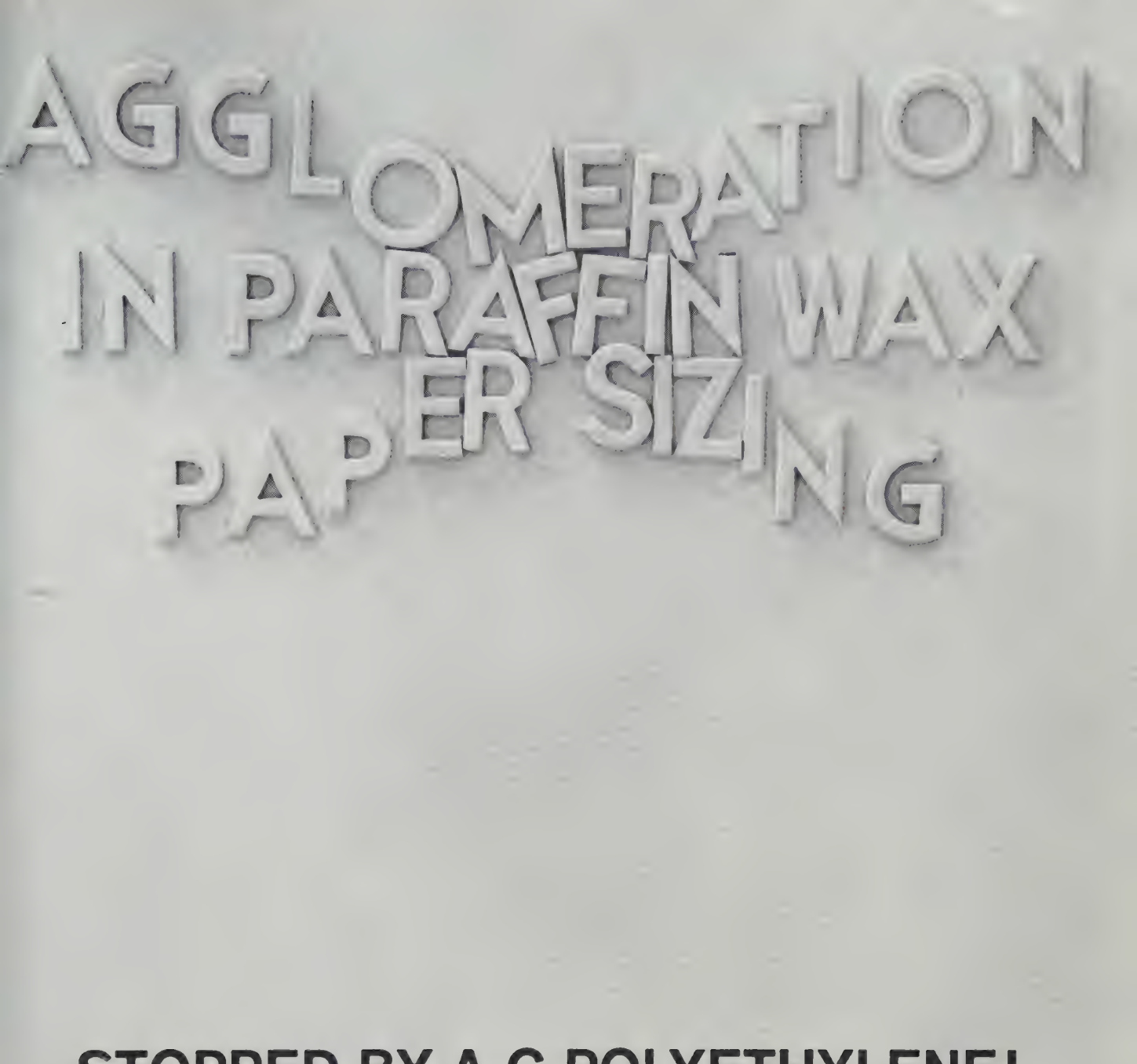

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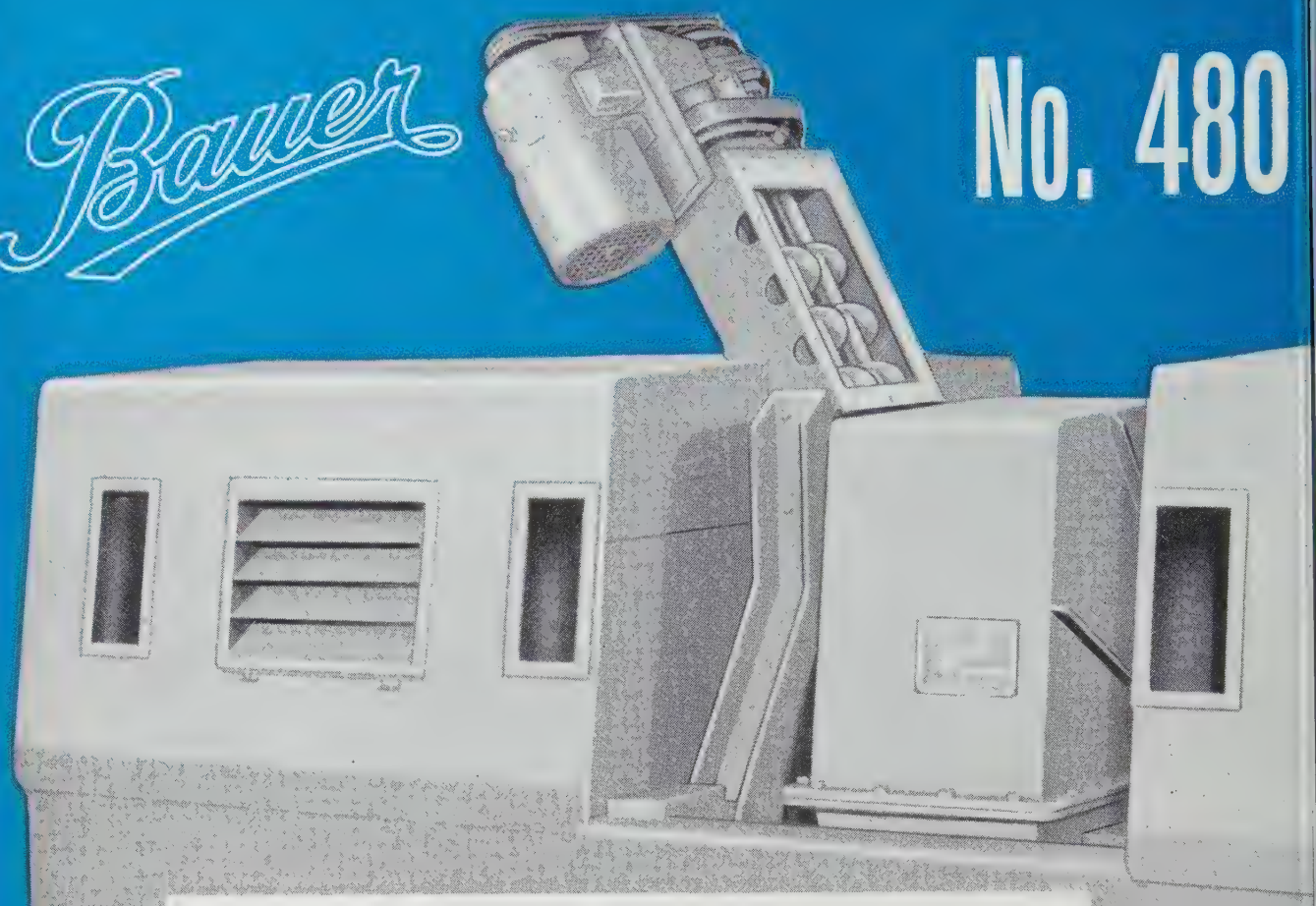

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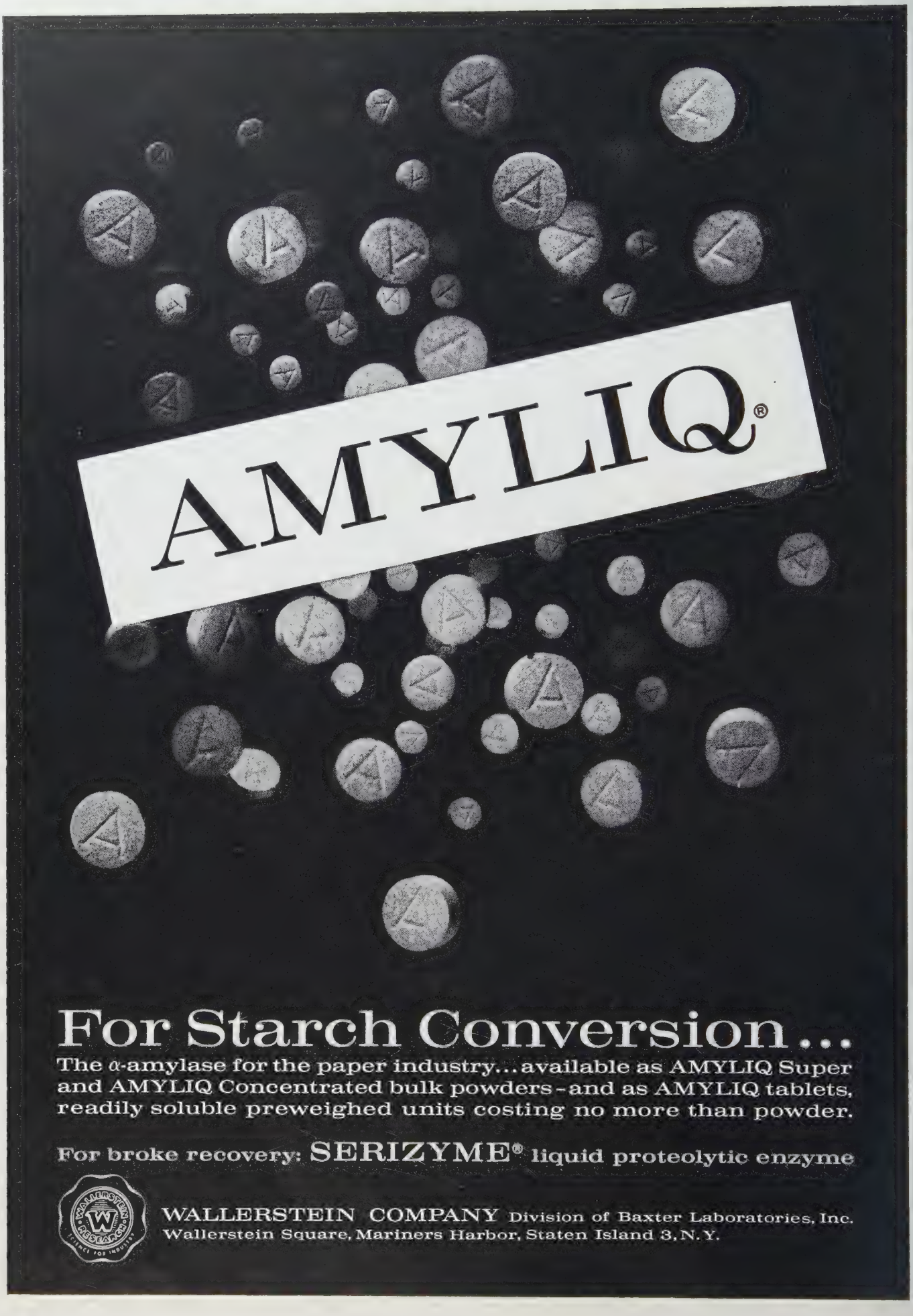
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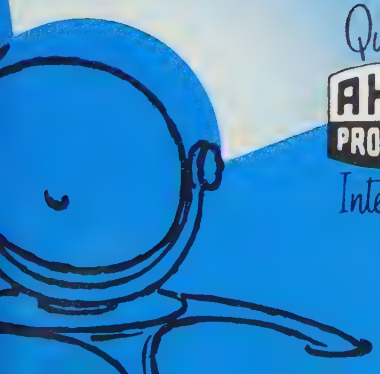

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Pulp Testing and Sheet Making Apparatus

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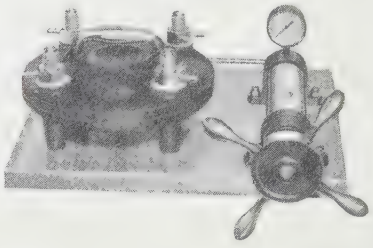

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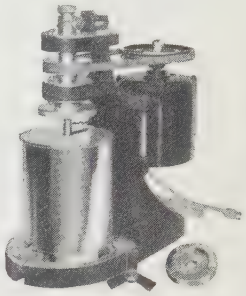

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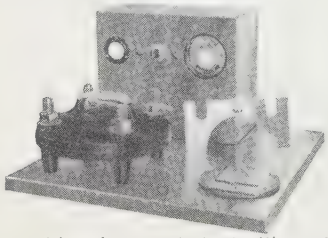

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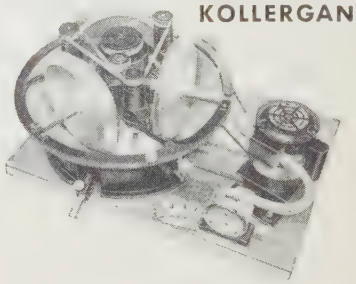

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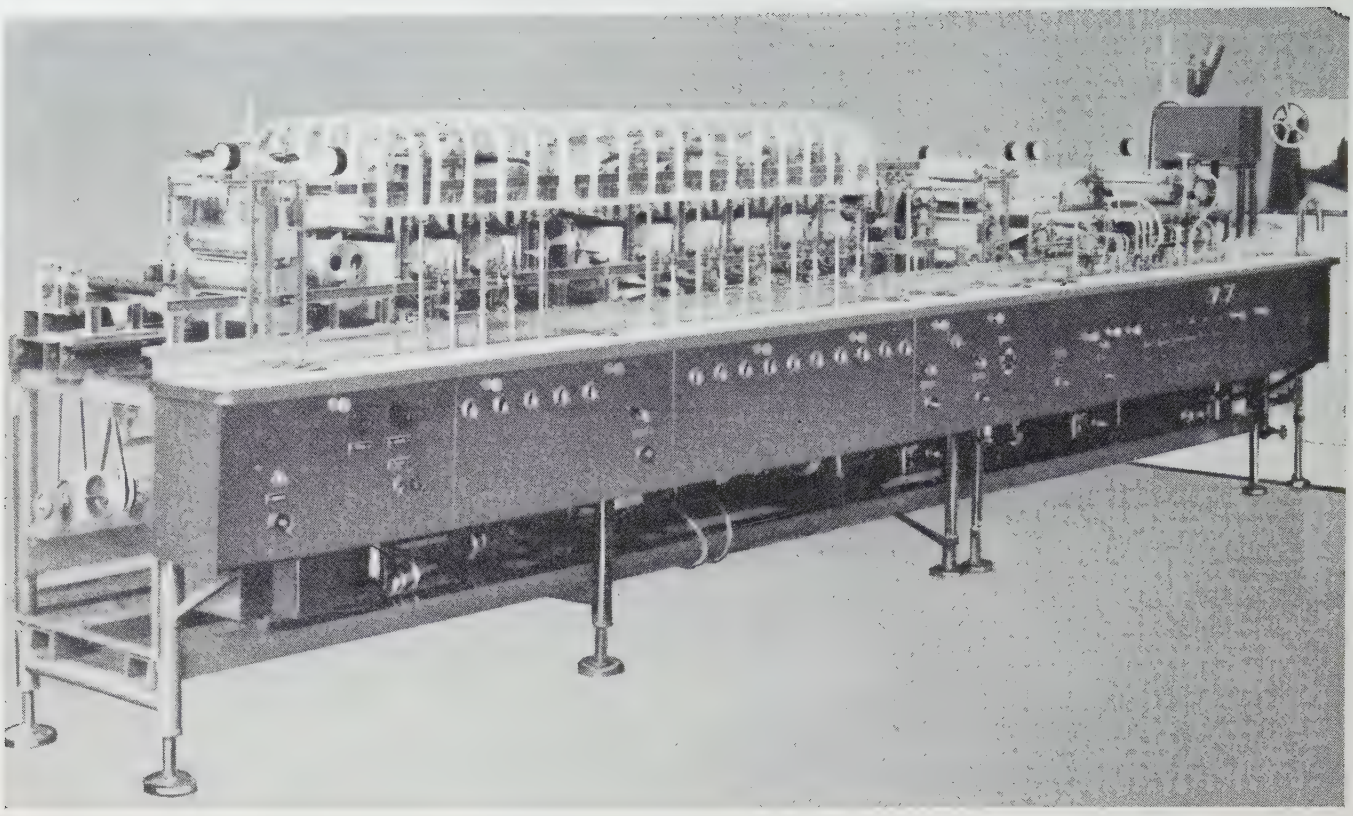
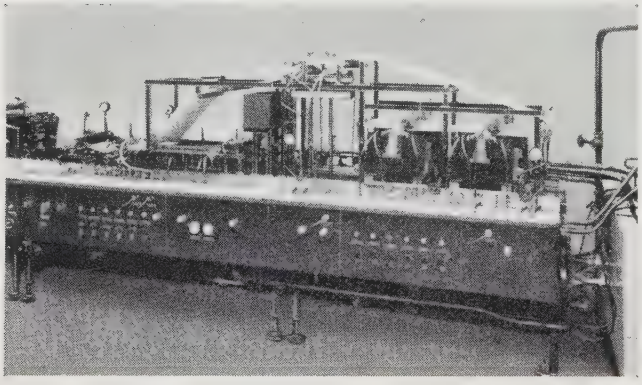


NEW P&W MIDGET FOURDRINIER PAPER MACHINE



The standard P&W Midget Fourdrinier model can also be operated in combination with miniature cylinder molds to provide more versatile sheet formation experiments.

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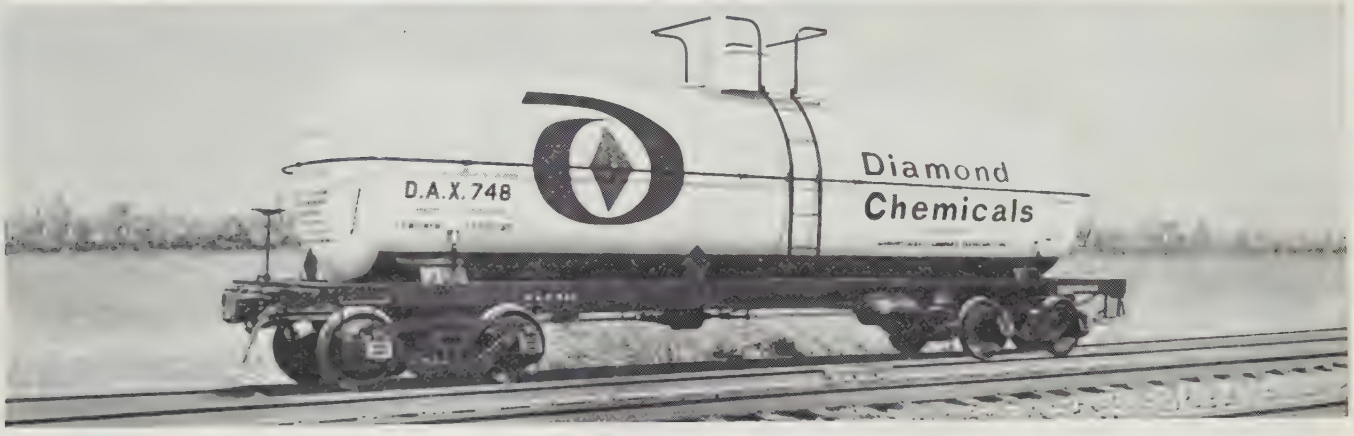
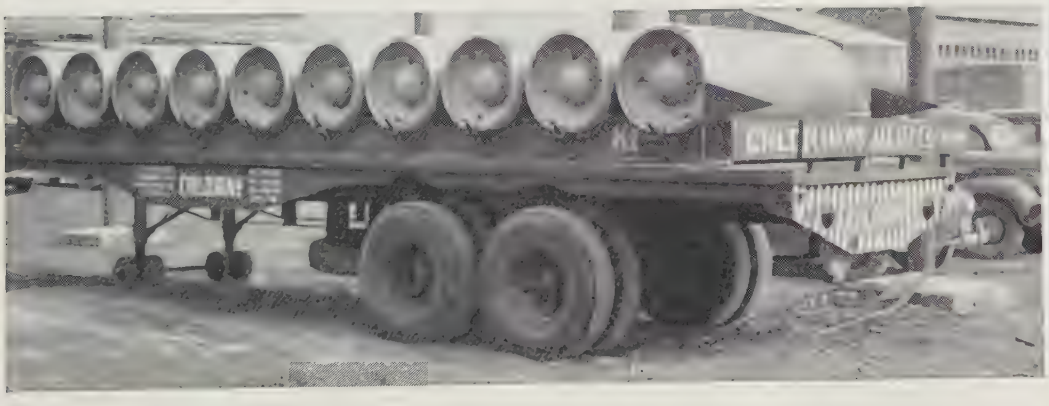


by tank cars...or by the cylinder or container



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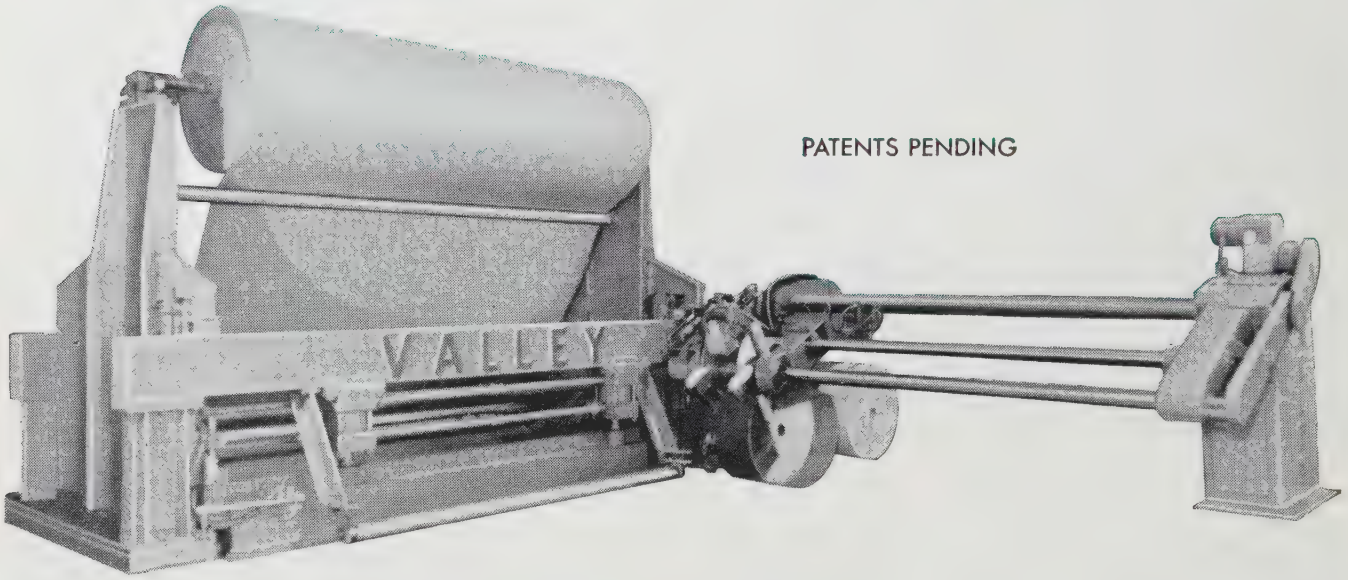

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Each phase of automatic time cycle can be individually activated by turning control console selector switch to manual, and pressing individual operation pushbutton mounted on the console board.

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Actual mill use is showing substantial savings in bleaching costs by the addition of Versenex® 80 chelating agent to groundwood pulp before bleaching. In some cases, brightness levels are maintained with less peroxide when Versenex 80 is used as a pretreatment. In others, gains in brightness are achieved without increasing peroxide concentration. In either instance, the cost per point of brightness is lowered.

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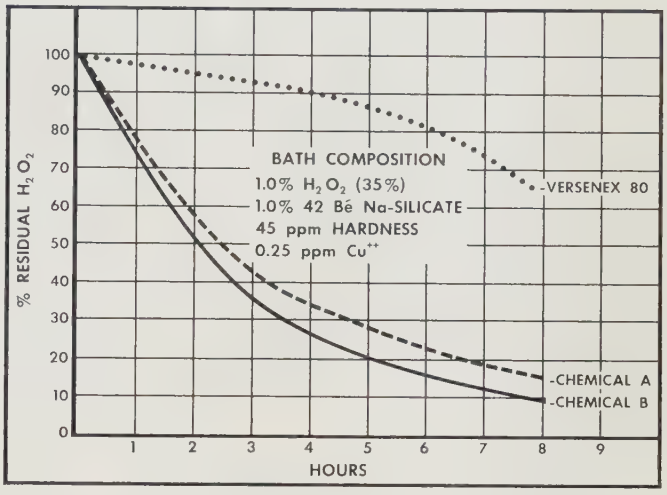
A comparison of Versenex 80 with other commercially available aminocarboxylic acids is shown in the graph at right. Versenex 80 pentasodium salt of diethylenetriaminepentaacetic acid is shown to be significantly superior to either tetrasodium salt of ethylenediaminetetraacetic acid (chemical A) or trisodium salt of N-hydroxyethylethylenediaminetriacetic acid (chemical B) for this application.

Where stray metal ions are causing reversion,



scaling, and other bleaching problems, investigate Versenex 80. For more technical information or for a trial run in your own plant, call your nearest Dow sales office or write us in Midland.

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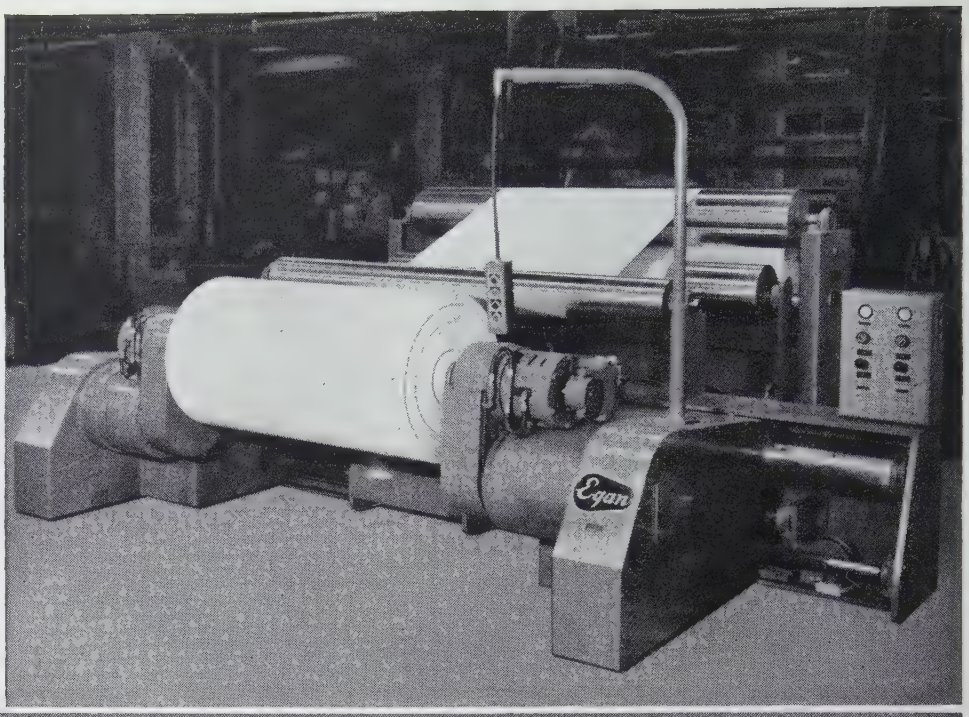
The unit shown at right was installed at the S. D. Warren Company's Plant, Cumberland Mills, Me. It operates at speeds up to 1000 fpm and handles rolls 84″ wide and 50″ in diameter.



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EGAN SHAFTLESS UNWIND

Egan presents the newest . . . most advanced . . . most compact . . . shaftless unwind ever designed for unwinding and converting applications!

The Egan Shaftless Unwind has eliminated the cross shaft between the pick-up arms. This saves considerable floor space by permitting the use of extremely short arms. As a result, the unit is more rugged; more compact, and allows for greatly reduced gear loading.

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The unit's greater rigidity also assures better operation at high speeds. An Egan Shaftless Unwind, which will handle rolls 12″ to 90″ wide with diameters ranging from 14″ to 60″, is capable of speeds up to 3500 fpm.

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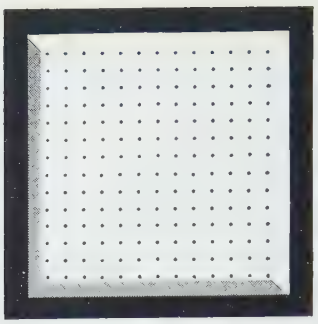

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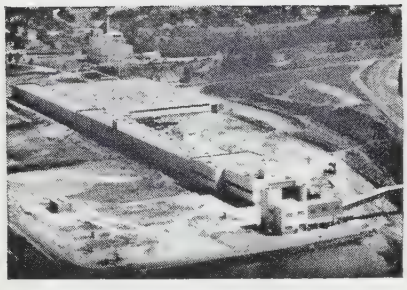
For the world's most automated fiberboard mill, CELOTEX specified complete mill instrumentation, Honeywell style—*pre-piped, pre-wired, installed, and serviced.*

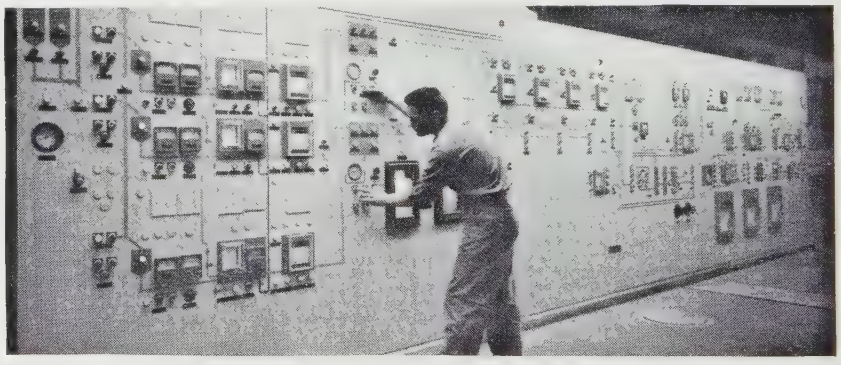
CELOTEX solved *with one Honeywell contract* the 1001 special instrumentation problems involved in getting the company's new L'Anse plant into production: instrumentation analysis of the fiberboard manufacturing process; supplying the complete control system; installing the control instrumentation with complete pneumatic piping and wiring; testing and calibrating the system; training CELOTEX personnel to operate and service the system; and providing complete guaranteed servicing for one year after completion of installation. *All in one complete package!*

Honeywell's *Pneumatic and ElectriK Tel-O-Set* recorders, transmitters, controllers, and other miniature instruments bring significant savings to this application. They not only save money and panel space but also permit consolidation of instruments into a more compact, easier-to-read graphic panel. All the instruments used in the pulping operation, for example, are grouped on a single panel rather than two or three which would be required if full size instruments were installed. By using both pneumatic and electric instruments in the control system, CELOTEX is able to utilize the inherent advantages of both types.

Find out more about *Complete Mill Instrumentation, Honeywell Style!* Get in touch with your local Honeywell field engineer. Be sure to ask him about the cost- and space-saving advantages of *Pneumatic and ElectriK Tel-O-Set* instruments. MINNEAPOLIS-HONEYWELL, Wayne and Windrim Avenues, Philadelphia 44, Pa. In Canada, Honeywell Controls, Ltd., Toronto 17, Ontario.



At the new L'Anse plant in Upper Michigan, The CELOTEX Corporation produces a variety of acoustical and decorative fiberboard ceiling tile, insulating sheathing, insulating roof deck, and roof insulation.



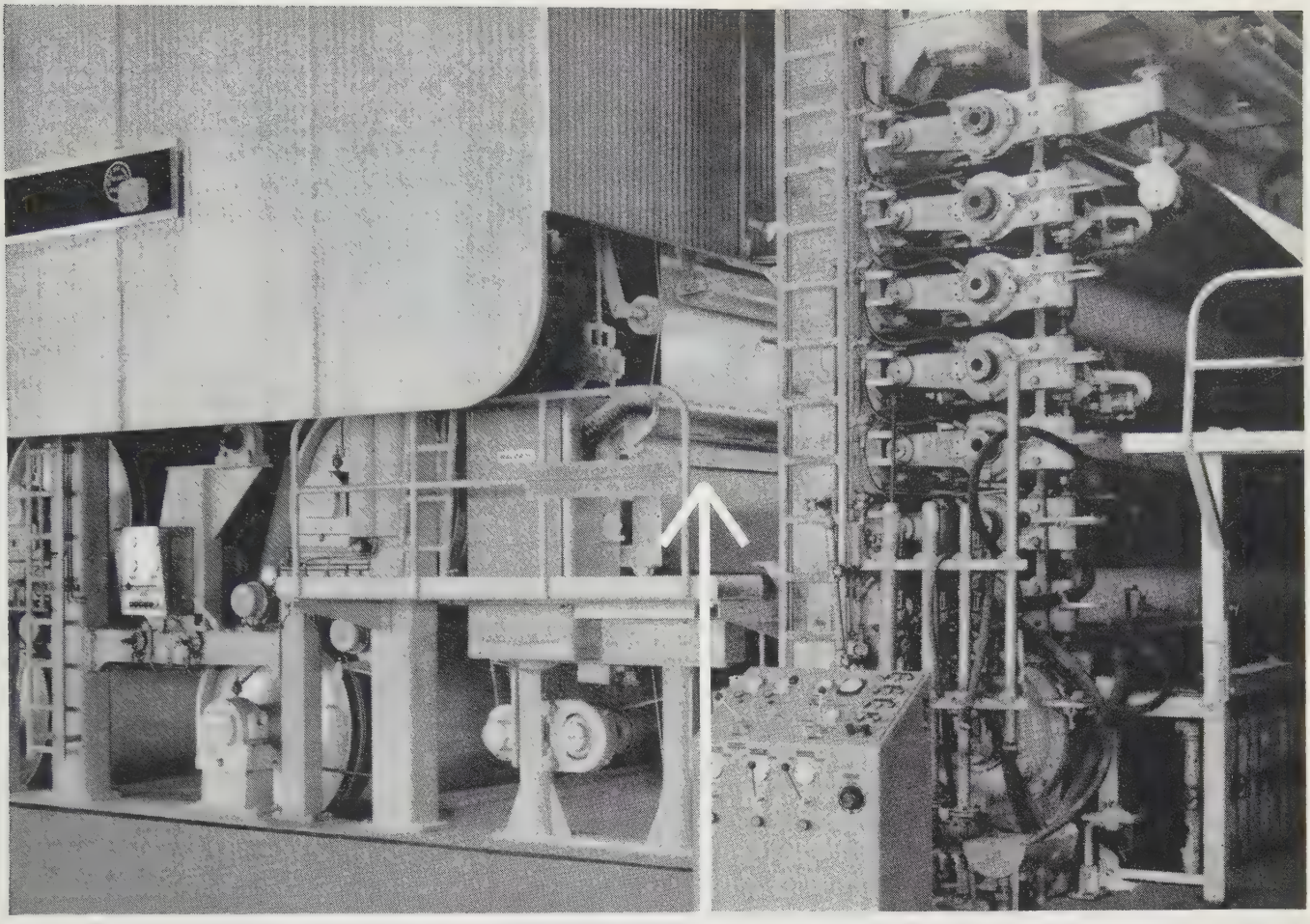
This graphic panel and other instrumentation, made, installed, and serviced by Honeywell for the L'Anse plant, help make this operation "the only one of its kind in which a fiberboard product travels all the way from manufacturing to packaging on a continuous production line."

Honeywell

First in Control

SINCE 1885

HONEYWELL INTERNATIONAL Sales and Service offices in principal cities of the world. Manufacturing in United States, United Kingdom, Canada, Netherlands, Germany, France, Japan.

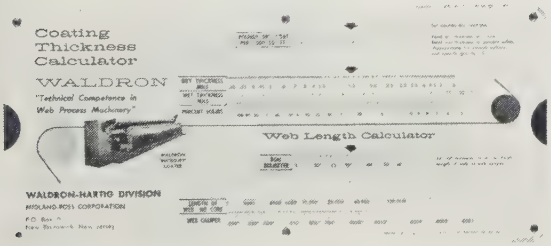


226″ Microjet® coater | production proven

On the paper machine shown above the Waldron Microjet coater provides an accurately metered film of coating for printability or functional properties on paper and board surfaces at high speed. The proven Microjet air doctor applies coatings on smooth pre-coated or rough uncoated surfaces with a higher efficiency than any other coating method and gives uniform and economic control of coating weight over a wide range. The coater is available for on-machine or converting operations, either as a unit or as a complete installation with all associated mechanical and Ross drying equipment.

The experience of more than a hundred machines in operation provides Microjet users with the best production-minded machine in the industry. For additional information write for bulletin 1010. Complete laboratory facilities in our plant for coating and drying commercial equivalent products are available to you.

Write for new convenient "Coating Weight Calculator". No obligation.



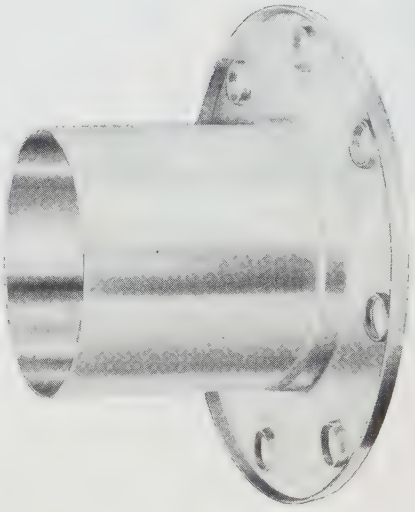
WALDRON

technical competence in web process machinery

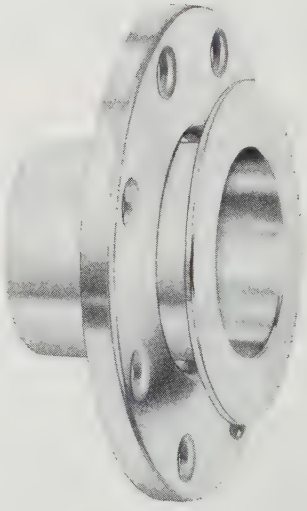
WALDRON-HARTIG, Box 791, New Brunswick, N. J./a division of Midland-Ross Corporation

NEW Lite-Wate* Flanges

* another unique development by FELKER



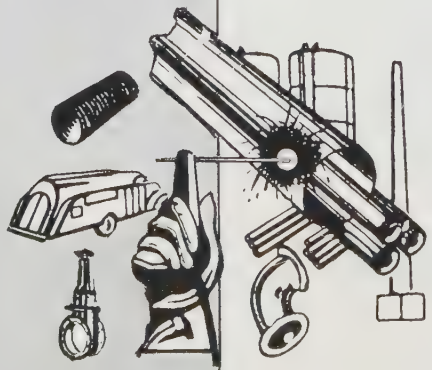
SLIP ON FLANGE
. . . weld on type



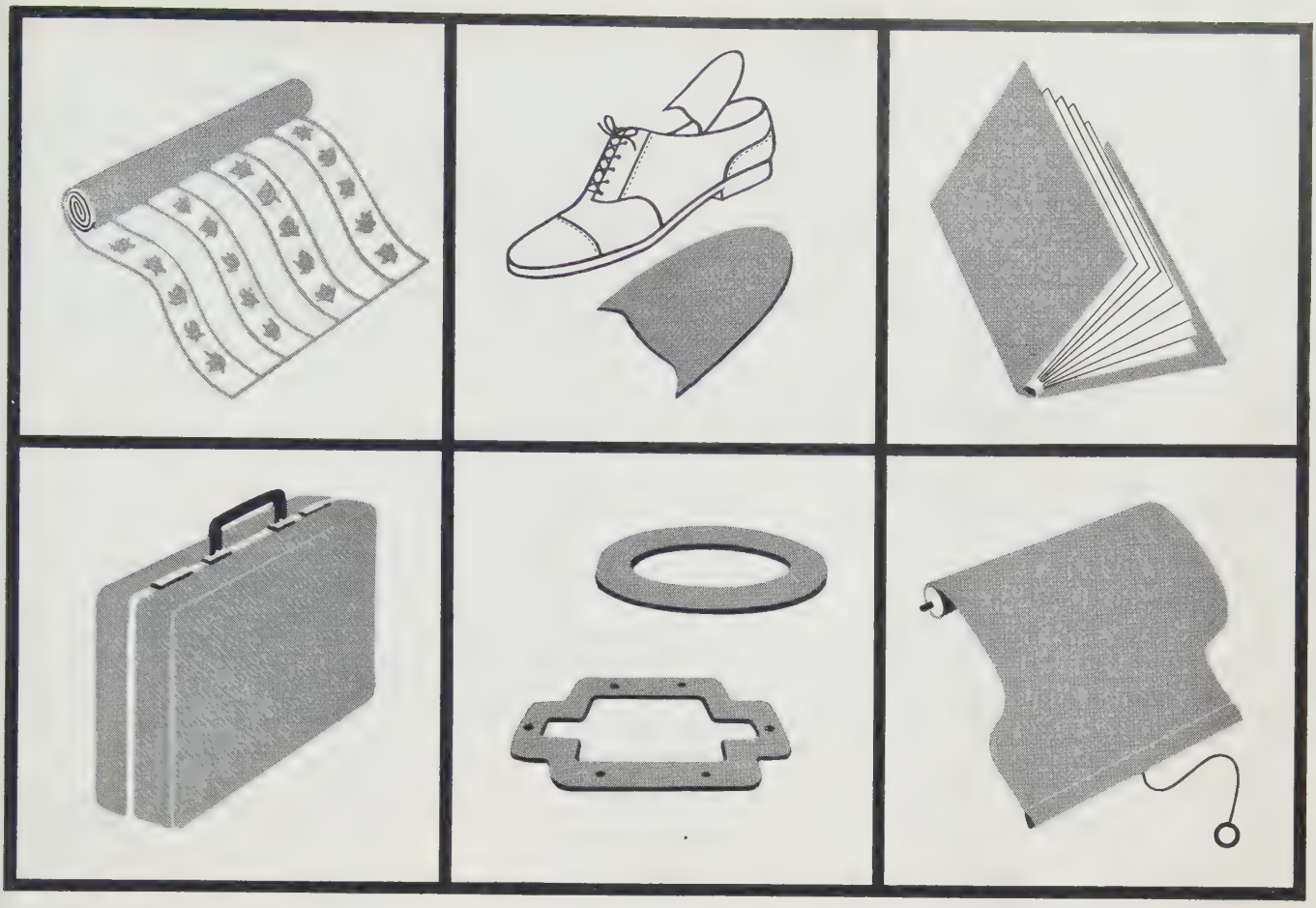
LAP JOINT
. . . back-up type

Write . . .

for bulletin number 46-47 which describes in detail LITE-WATE FLANGES presented to the industry as a result of progressive thinking, development and research.



FELKER BROS.
MANUFACTURING CO.
MARSHFIELD, WISCONSIN
*Phone: FUlton 4-3121 (area code 715)
*TWX: Marsh 8912



Produce saturated papers or moldable forms with Rhoplex® acrylic emulsions

Leather-like papers, papers with a fabric-like feel, papers with weather-resistance, paper sheets with thermoplastic moldability—you can produce all these and many variations, by saturating paper with RHOPLEX 100%-acrylic emulsions.

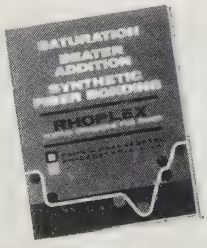
A series of RHOPLEX polymer emulsions is available. The polymers range from hard, thermosetting types to soft, yielding ones. These emulsions may be used individually, or blended to produce combinations of their individual properties.

By using the appropriate polymer emulsion or combination of emulsions, properties such as the following can be imparted to paper: resistance to grease, oil, solvents and water; improved tear and tensile strength; toughness; and fold and delamination resistance. These properties are retained on aging and exposure to a wide range of conditions as well.

Possible uses for papers saturated with RHOPLEX emulsions include floor and wall coverings, shoe and heel counters and inserts, cover stock for embossing, water-resistant sandpaper backing, artificial leathers, window shade materials, gaskets, and molded panels for auto interior trim.

In addition to providing advantages in the finished product, RHOPLEX emulsions offer processors exceptional mechanical and chemical stability. They are compatible with many other latices, and can be chemically modified to produce a number of different useful effects.

Complete technical information on these versatile, useful acrylic emulsions is available on request.

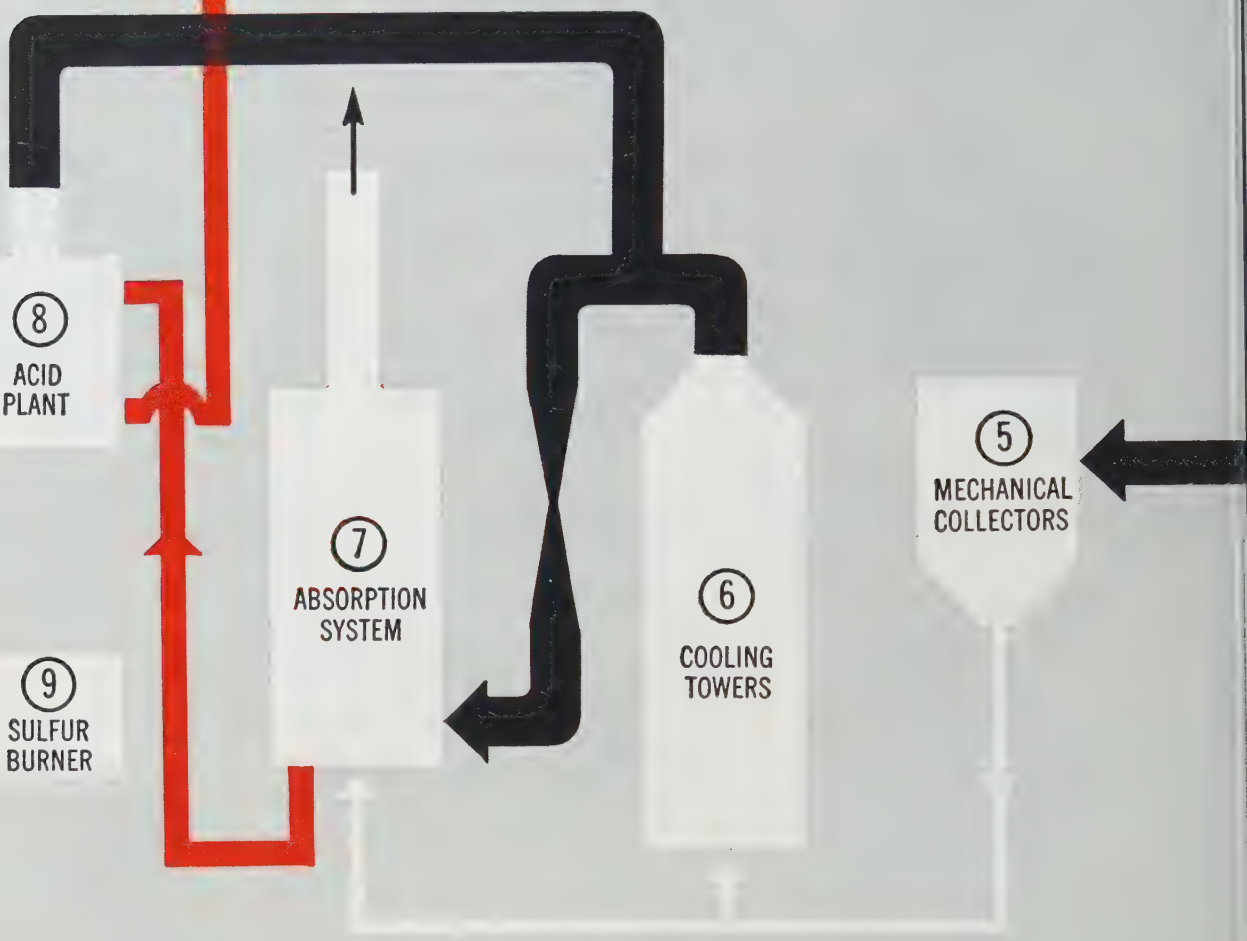


Write Dept. RP-1 for a copy of this new booklet describing RHOPLEX *resins for saturation applications, beater addition, and fiber bonding.*

ROHM & HAAS
PHILADELPHIA 5, PA.

RHOPLEX

MgO pulping and recovery process. Pulp wood chips are digested in magnesium bisulfite liquor (1) pulp is withdrawn and washed (2) and knots removed. Liquor with 10 to 15% solids is passed through multiple-effect evaporators (3) to raise the solids content to 55% before burning in recovery boilers (4) to produce steam at 860 psi and 825 F for power generation and processing. MgO and SO_2 are released from the liquor during the incineration process. MgO is recovered in mechanical collectors (5) while the SO_2 and flue gases pass to the cooling and absorption towers (6). Recovered MgO is hydrated and introduced into the acid circulating to the absorption system (7). Here it combines with SO_2 to form magnesium bisulfite acid. The cycle is completed by fortifying the recovered magnesium bisulfite acid (8) with sulfur dioxide from the sulfur burner (9).

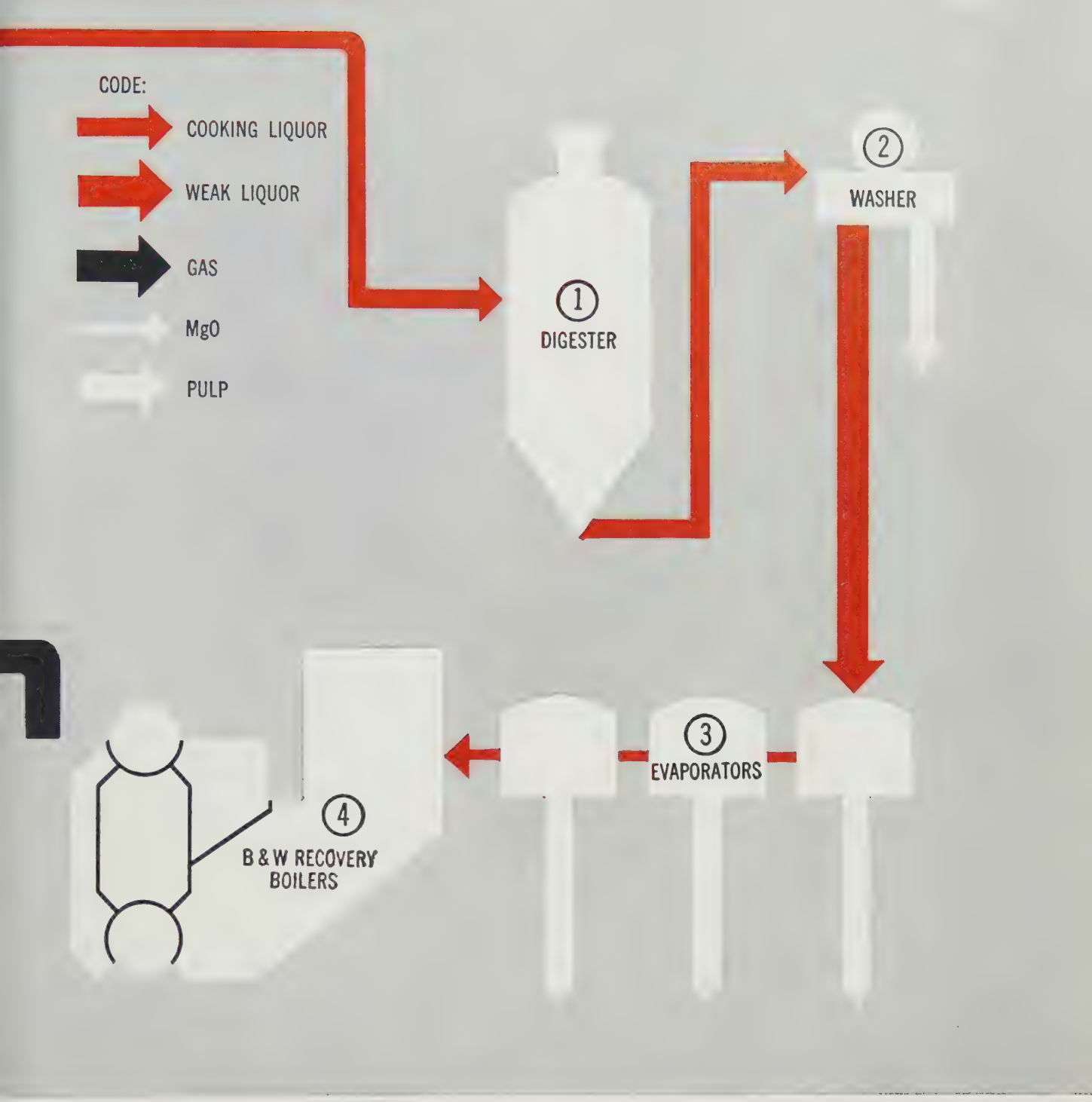


MgO process at Weyerhaeuser's Cosmopol

Evaporators in series increase pulping liquor from less than 15% to about 55% solids to permit its burning.

B&W MgO recovery boilers burn the concentrated liquor, supplying 70% of the plant's steam requirements.

Mechanical collector reclaims MgO from furnace flue gas and washes it before further use.



lill recovers 90% of spent liquor solids

Only 20 pounds of magnesium oxide and 40 pounds of sulfur per ton of unbleached pulp are needed for make-up at Weyerhaeuser Company's 400 ton per day sulfite mill at Cosmopolis, Washington.

The simple, closed cycle MgO pulping and recovery process, placed in operation during 1957, reclaims about 90% of the pulping chemicals. The B&W MgO recovery boilers provide 70% of the steam required for the entire plant's power generation and processing needs. A third B&W bark and oil fired boiler provides the balance of steam requirements.

The commercially-proven MgO pulping and recovery process is ideal for either new mills or conversion and modernization of existing mills. Shorter cooking time required for MgO pulping cuts capital and maintenance costs in new installations and permits increasing the production of existing mills. Quality sulfite pulp with high brightness and strength is produced.

For more information about the efficient MgO pulping and recovery process, contact your nearest B&W sales office or write The Babcock & Wilcox Company, Boiler Division, Barberton, Ohio. E-302-1001

Babcock & Wilcox

Nopcote® C-104 is out of this world when it comes to lubricating protein, starch or latex coating colors. A fluid-stable, 50% calcium stearate dispersion, it produces a clean, dust-free coating with improved gloss—even at low calender pressures.

This lubricant improves flow and leveling characteristics to the point where a smooth, even application is possible at high speeds and on any equipment—trailing blade, roll train, air knife, or size press. It minimizes surface pattern, too, and gives your sheet that extra increment of salability.

This mill-proven coating additive is fluid and easy to handle. Other advantages are that it has little effect on color viscosity and permits application of high-solids, quick-drying coatings.

Nopco technical representatives will be happy to supply samples of Nopcote C-104 and set up a mill trial at your convenience.

Dispersants
Defoamers
Wax Sizes
Calender Stack Lubricants
Detergents
Emulsifiers

NOPCO CHEMICAL COMPANY

NOPCO®

60 Park Place, Newark, N.J.

Plants: Harrison, N.J. • Carlstadt, N.J. • Richmond, Calif. Cedartown, Ga. • London, Canada • Mexico, D.F. • Corbeil, France • Sydney, Australia

Manufacturing Licensees Throughout the World

SCAPA DRYER FELTS

FIRST TO LAST

Famous Paper Mills of the World Serviced by SCAPA

BOWATER'S NEWFOUNDLAND PULP AND PAPER MILLS LTD.

Corner Brook, Newfoundland

FM-8

SCAPA DRYERS, INC. WAYCROSS GEORGIA

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Marey Paper Mill Supply Company
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John B. Chandler Co.
P. O. BOX 1748, ATLANTIC BEACH, FLA.

Tipka Supply Company
415 JACKSON ST., OREGON CITY, ORE.

VORTEX PULP CLEANER TIPS. Where abrasive wear is a problem, "KT" Silicon Carbide tips provide a minimum of 2 years service where previous life was 3 to 5 months.
SUCTION BOX COVERS. Wire wear is reduced as much as 40%... couch power and wire tension is reduced and resurfacing is virtually eliminated.
WEAR SLEEVES. In continuous pulp digester systems, "KT" Silicon Carbide sleeves outlast normal materials, such as high carbon steel, as much as 20 to 1.

Super-hard, corrosion-resistant, service-proved!

"KT" SILICON CARBIDE

"KT" Silicon Carbide, developed by Carborundum, is the hardest material *now commercially available* in large, complex shapes. Many problem areas in the processing systems of the paper, paperboard and fiberboard industries are being eliminated with this new, more durable material. Highly resistant to abrasion because of its self-bonded, dense structure . . . virtually impervious to corrosive attack by mineral acids at all concentrations and temperatures, "KT" also can be precision ground to high polish finishes. New application areas now being developed: orifices, felt conditioner shoes, valve ports for acid service, wear plates, spray nozzles, forming boards, wire guide pans, coal pulverizer parts and black liquor nozzles. *For more specific details* or for engineering assistance, write New Products Branch, The Carborundum Company, Niagara Falls, New York. **CARBORUNDUM**

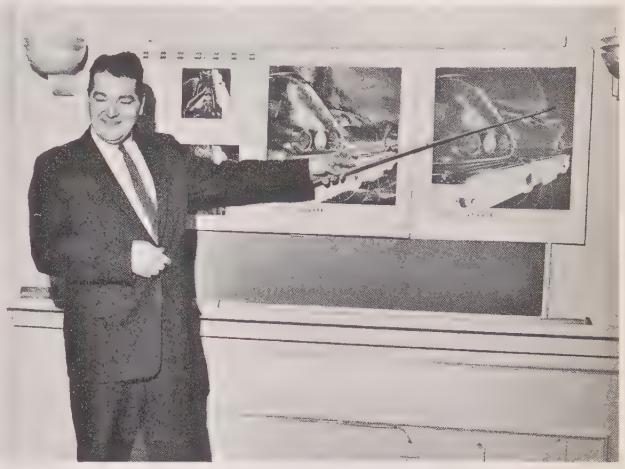
BARNES BARNSTORMS U. S. WITH COLORED PAPER PROGRAM

The gentleman pictured here in action is Roy M. Barnes, sales promotion manager for the Du Pont Dyes & Chemicals Division and tireless exponent of the creative use of colored paper by graphic artists.

As spokesman for Du Pont's colored paper promotion program, Barnes has traveled thousands of miles and talked to an estimated 9600 artists, printers and paper industry personnel in the past three years. He has told them about Du Pont's survey of the graphic arts (conclusion: an unrealized potential exists for colored paper sales), shown them how colored paper is being used functionally and impressively by artists, described how paper makers are cultivating the colored paper market.

In one recent week, for example, Barnes flew from Delaware to Grand Rapids, Michigan, to speak to an audience of printers and artists. The next day found him in Kansas City addressing a group of paper merchants, paper manufacturing personnel, artists and printers. Thence by air to Rochester, N. Y., where he addressed two groups—a meeting of paper merchants and a printers' organization. Tally for the week: 3200 miles traveled, 1150 persons talked to, one very weary Barnes.

Five other Du Pont men, working out of San Francisco, Chicago, New York, Philadelphia and Providence, also are engaged in telling the colored paper story. If you're interested in booking Du Pont's colored paper presentation for a meeting at your company or if you'd simply like to know more about it, we'll be delighted to oblige. Address your inquiry to the Du Pont Color Council, N-8421, Wilmington 98, Delaware.



news from Du Pont

NEW WEEKLY DU PONT SHOW ON NBC-TV

A new television program, the "Du Pont Show of the Week," debuts September 17 to an estimated weekly audience of 19 million viewers.

The program, scheduled from 10 to 11 p.m., EDT, Sundays, over NBC-TV, will deal with actual people, situations and events. Included will be musical variety programs, drama based on real life, and special projects on matters of national importance. The new program takes the place of the former "Du Pont Show of the Month" and "Du Pont Show with June Allyson."

Both corporate and product advertising will appear on the program. Among the Du Pont products to be advertised are textile fibers, paint, antifreezes, car-care products and coated fabrics.

NOW: A WHITE TO KEEP YOU OUT OF THE RED

Interested in saving money in your use of fluorescent white dyes? We ask this rhetorical question by way of introducing as a suggested answer the latest in Du Pont's line of optical brighteners—Paper White SPS Solution.

SPS works well in the beater, but it's especially formulated for surface application. And surface application is the cheapest way of whitening a sheet with a basis weight of 16 pounds or more. If you haven't done it already, get our laboratories to review your present method of applying whites with an eye to suggesting economies.

SPS is also available in a 50-cents-a-pound diluted concentration called Paper White SPSW Solution.

DU PONT
REG. U. S. PAT. OFF.

DYES AND CHEMICALS

Better Things for Better Living
. . . through Chemistry

E. I. du Pont de Nemours & Co. (Inc.), Organic Chemicals Department, Dyes and Chemicals Division, Wilmington 98, Del.

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Portland 9, Ore. . . . . 1238 N. W. Glisan St.
Rumford 16, R.I. . . . . 50 North Broadway
DU PONT OF CANADA LIMITED, Montreal, Canada

MAJOR MILL NETS AN AVERAGE OF $4/TON SAVINGS IN WIDE RANGE OF PAPER SHADES... THANKS TO "ECONOMY CHECK"

One of our district offices is now finishing up an Economy Check on 110 production shades for a progressive paper mill in the area. Already, with about 70% of these shades analyzed, Du Pont's extensive evaluations have yielded significant results:

1. Average cost savings of about $4 per ton of paper by substituting cheaper dyes without important losses in quality.
2. Some shade quality upgraded by alternative dyes, in many cases at no extra cost.
3. Mill efficiency improved by new dyes that are easier to control, give more uniform results, are more adaptable to the paper being produced.

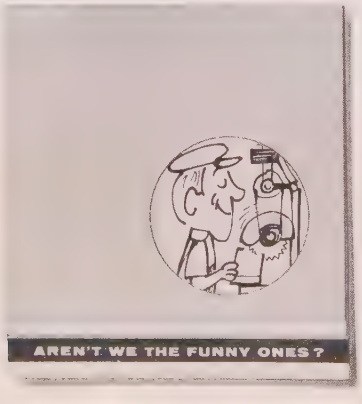
The following table gives actual examples of the five shades being run most frequently by the mill, with cost and qualities both before and after Du Pont recommendations. If you haven't had *your* Economy Check, be sure to ask for it from your local Du Pont representative.

SHADE	COST OF OLD PRODUCTION FORMULA	COST OF NEW FORMULA	REMARKS
Buff	$11.22/ton Four colors	$7.90/ton Two colors	Properties of new formula similar to old. Previous formula not bleachable; present formula fully bleachable.
Yellow	$17.50/ton Two colors	$7.22/ton Two colors	Old and new formulas fully bleachable. Lightfastness of new somewhat inferior to old. This reduction in physical property can be tolerated in particular grade of paper.
Green	$3.22/ton Two colors	$1.68/ton Three colors	Previous formula non-bleachable. New formula fully bleachable. Lightfastness slightly reduced. Third color in new formula improves sides considerably over old.
Blue	$42.02/ton Three colors	$13.02/ton Three colors	Physical properties identical to old, with improvement in lightfastness stability. Both formulas bleachable to within 90-95% of total color destruction.
Red	$8.95/ton Four colors	$4/ton Two colors	Slight reduction in lightfastness with new formula. Properties considered entirely satisfactory for bond shades.

news from Du Pont

FREE BOOKLET FUN TO READ

"Aren't We the Funny Ones?", a cartoon booklet on the subject of foreign competition in the American market, is now in its fourth printing. The booklet is a product of the Du Pont Dyes & Chemicals Division. For your free copy, write to the division, Nemours 8421, Wilmington 98, Del.



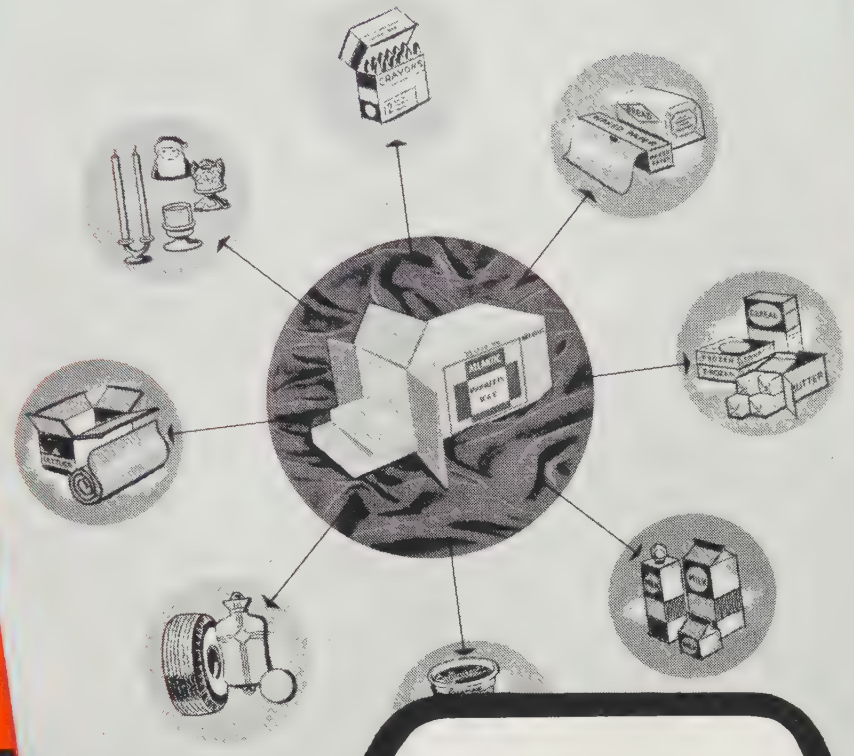
"ZEPAR"® BP BLEACHING AGENT —GOOD BRIGHTNESS, LOW COST

Producers of groundwood pulp should take a long, hard look at the Du Pont bleaching agent developed specifically for their product — "Zepar" BP reducing agent. It produces a high degree of brightness at minimum cost and without loss in pulp weight.

"Zepar" BP increases groundwood brightness by as much as 10 to 14 GE points. For even greater brightness, you can bleach with peroxide first. For a 20-point brightness increase, try an initial bleach with Du Pont "Albone"® 50 hydrogen peroxide, "Solozone"® sodium peroxide, or a combination of the two, followed by a bleach with "Zepar" BP. The peroxide accounts for a 12-14 point increase, while an additional 7-point boost comes from "Zepar" in the second stage.

To repeat a familiar admonition: Ask your Du Pont salesman for more details or call the nearest district office.

AN ATLANTIC WAX FOR EVERY USE



FREE for your files...

A helpful NEW 24-page guidebook for all users of paraffin waxes

Whatever your individual requirements, you're sure to find information of value to you in this new Atlantic guidebook. This is because Atlantic offers a complete line of waxes covering every industrial need.

The booklet describes petroleum waxes and their uses... discusses wax properties such as gloss, grease resistance and sealing strength... provides a handy chart of recommended waxes for specific purposes from acid bottles to woodwork finishing... and more!

Of particular interest is the section devoted to waxes and types of waxing used in the paper coating industry. Here, for example, you will find the proper blend for a hard, glossy surface, advantages of waxed corrugated cartons over wooden crates, and descriptions of wet, dry and laminated waxing operations.

To receive your free copy of the booklet, simply fill in and return the coupon below. And if you'd like more detailed information on any Atlantic wax or blend, get in touch with your Atlantic Sales Engineer. No obligation, of course.

THE ATLANTIC REFINING COMPANY
Industrial Products Division
260 South Broad Street, Philadelphia, Pa.
Please send me—without obligation—a copy of your new 24-page guidebook, "An Atlantic Wax for Every Use."

Name_____

Title_____

Firm Name_____

Address_____

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WAXES • LUBRICANTS • PROCESS PRODUCTS

Philadelphia, Pa. • Providence, R. I. • Syracuse, N. Y.
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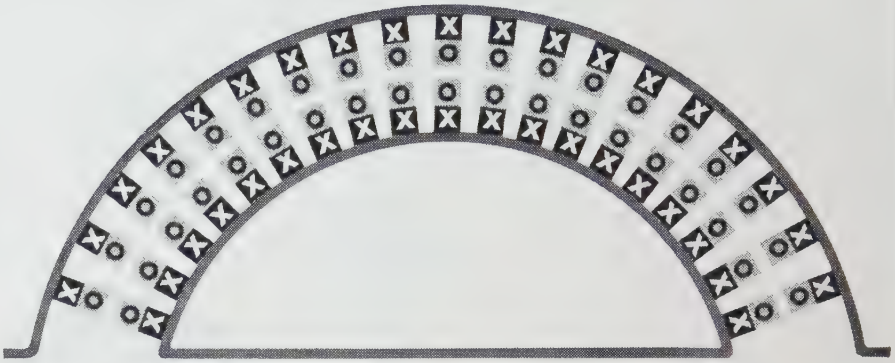
ATLANTIC

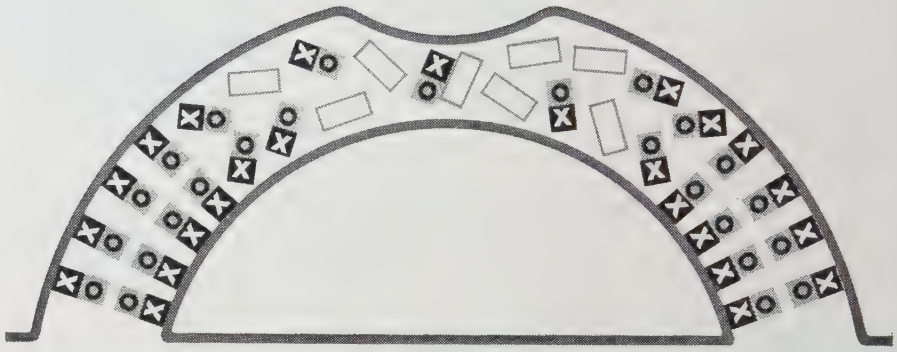
The make-up and break-up of foam

Foam, of course, is made up of bubbles. But, all bubbles do not form foam. For instance, pure water will not foam. If you take a glass or a beaker of pure water and bubble air through it, the bubbles will be large and will rapidly rise to the surface and burst. Several smaller bubbles may even join together and rise to the surface. But even these burst at the surface. There is no tendency to form foam.

Now, add just one drop of detergent—a surface active material—to this same water, blow air through it and foam develops. The bubbles are relatively small and there is no tendency to coalesce. This foam-causing difference is due to the surface activity of the detergent molecules.



Bubble Cross-section



Bubble with Defoamer Added

The Make-Up of Foam

Each surface-active molecule has a "water-loving" end and a "water-hating" end. These molecules arrange themselves in an orderly fashion in the liquid film of the bubble with their "water-hating" ends toward the film surfaces and their "water-loving" ends buried in the liquid interior of the film. It is this arrangement of molecules that gives strength and elasticity to the bubbles and keeps them from bursting.

The Break-Up of Foam

Anti-foam agents act either by completely displacing the surface film or by modifying its film-forming properties so that it no longer forms a stable foam. Therefore, a good defoamer must be capable of entering the liquid interior of the bubble film and modify or break-up the molecule arrangement that sustains the bubble film. Thus, since the defoamer only has to work on only a small quantity of surface active material, only a relatively small amount of defoamer is needed.

What Causes Paper Mill Foam?

Some impurity, intentional or otherwise, must be present before foam can be produced. The most persistent foams are produced when the impurity is a surface active material such as carbohydrates, lignin, dyes, black liquor soaps, slimicides, sizes, etc. Since a foam condition can be caused by any one—or several—of these materials, no single defoamer will fill all needs economically. Houghton's "Personalized Paper Mill Service" was instituted to provide this "diagnosis and curative" service with proven De-Airex defoamers. If you have a foam problem in your mill, call your Houghton man or write and request this technical help. It's yours for the asking and may save you untold headaches and expense. E. F. Houghton & Co., 303 W. Lehigh Ave., Philadelphia 33, Pa.

Houghton

INDUSTRY'S PARTNER IN PRODUCTION

from DeZurik... A GREAT NEW

COMPACT PIPELINE CONSISTENCY REGULATOR

REFLECTING 36 YEARS
OF
DeZURIK EXPERIENCE
AND LEADERSHIP IN
CONSISTENCY
REGULATION

There's a great pedigree behind this notable new Pipeline Regulator. It bears the reputation and reflects the know-how established by the many thousand DeZurik Regulators in successful service around the world.

This new Pipeline Regulator is remarkably compact without sacrificing one fraction of DeZurik's excellence and competence. It installs right in line on the line, requires virtually no extra space, needs no extra supports. Installation is easy, quick, trim, economical.

THIS IS THE ONE COMPACT REGULATOR THAT OFFERS ALL THE ADVANTAGES OF DeZURIK'S MOTOR-DRIVEN SENSING ELEMENT! Through millions of hours of use, the DeZurik rotating sensor has clearly demonstrated its freedom from fouling and from stock hang-up. Even the stringiest stock is controlled accurately without the plug-ups occuring on stationary feelers. No shut-downs, practically no maintenace.

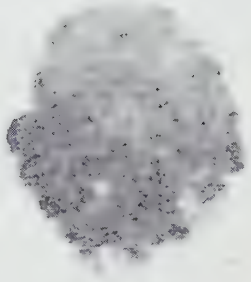
DeZURIK'S FAMED ACCURACY AND SENSITIVITY are guaranteed in this compact unit. It will positively hold consistency within plus-or-minus .1%; its proved performance is closer to a variation of ONLY .02%!

The DeZurik Compact Pipeline Regulator is a COMPLETE regulator, with the finest recorder-controller instrumentation, adaptable readily to your specific requirements. Full data and costs on request.

DeZURIK CORPORATION
SARTELL, MINNESOTA
Since 1925 — A Partner Of The Paper Industry

NEW AIRCO VINOL-BORAX SIZING

REDUCES PENETRATION OF OILS AND GREASES

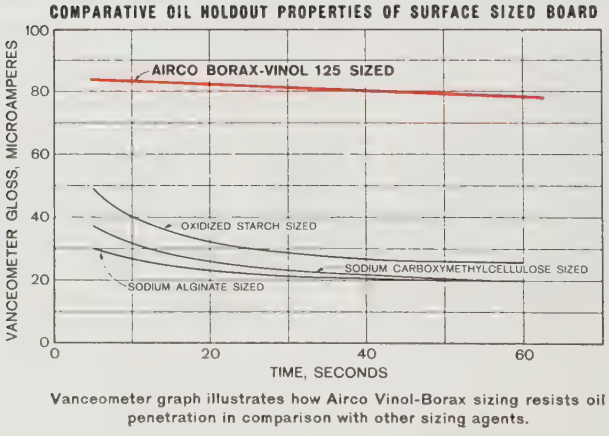


STARCH SIZED SHEET

OIL ABSORPTION TEST

Five drops of NCS-12 oil were dropped from a dropper and allowed to stand for 60 seconds and then photographed from the back. The result shown here tells the story.

VINOL-BORAX SIZED SHEET



Vanceometer graph illustrates how Airco Vinol-Borax sizing resists oil penetration in comparison with other sizing agents.

THE new Airco Vinol-Borax sizing process can cut costs as much as 45% in mill operations. Its superior performance, demonstrated in the laboratory test shown above, is amplified in actual use.

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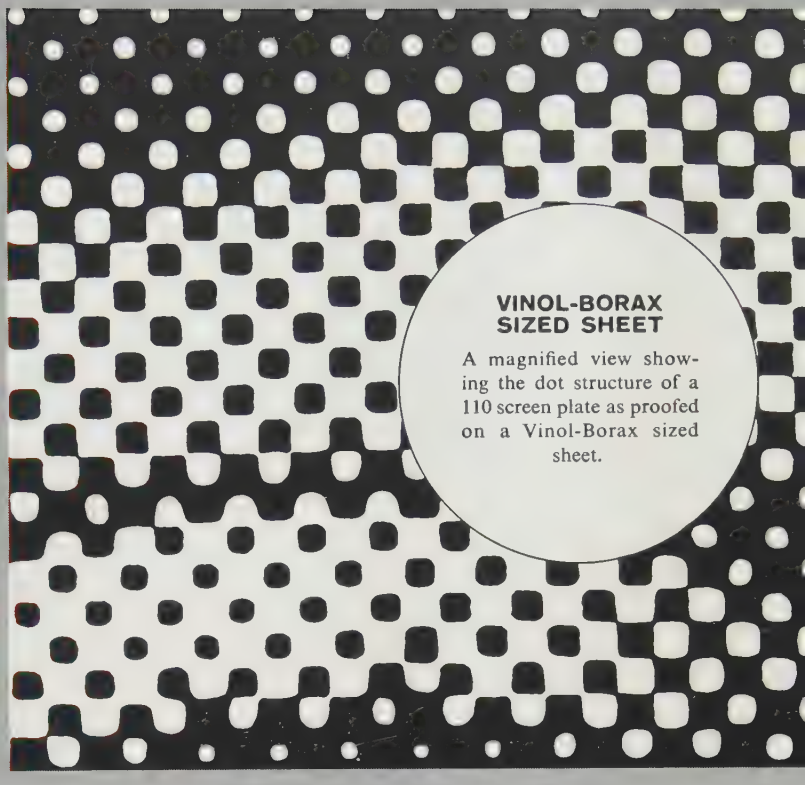

VINOL-BORAX SIZED SHEET

A magnified view showing the dot structure of a 110 screen plate as proofed on a Vinol-Borax sized sheet.

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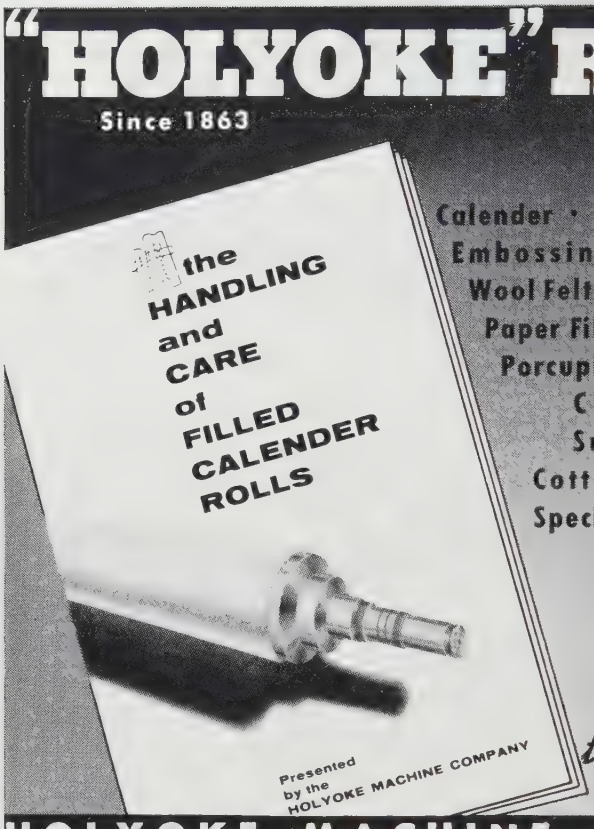

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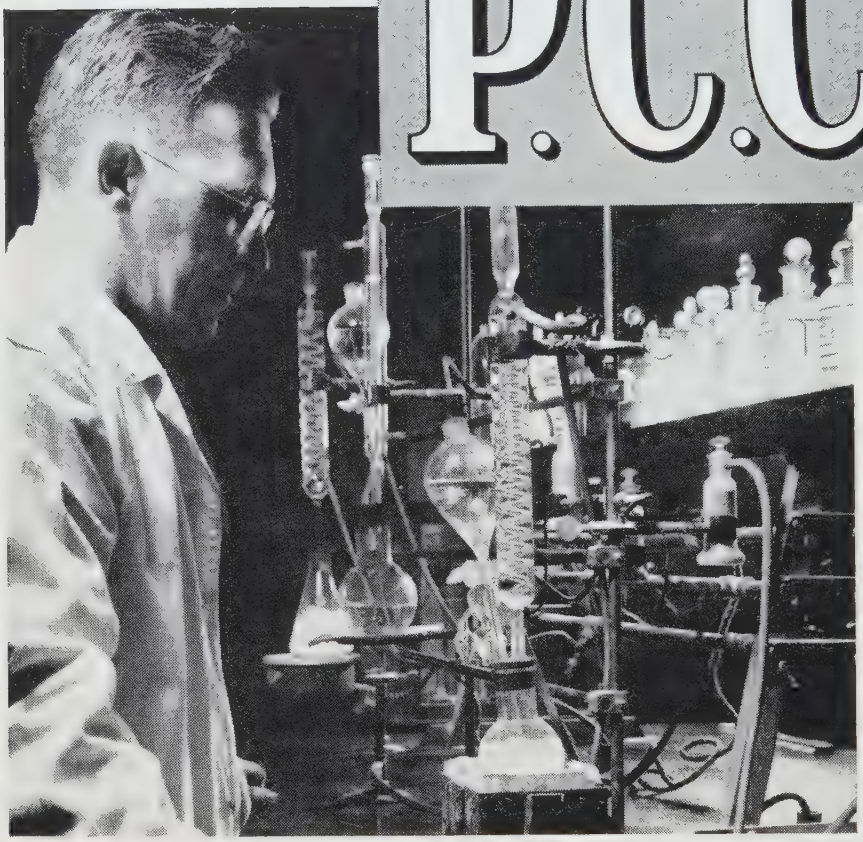

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The Kopparfors Bag Type Digester Lining

Stress Analysis and Engineering Details

G. CAPE and A. M. BAIN

The Kopparfors bag lining is a method of protecting the inside of digesters operating under cyclic conditions of temperature and pressure, whereby large thin sheets of suitable corrosion resistant metals protect the inside surfaces of the carbon steel. The lining is attached to the vessel at nozzle liners and two or more girth joints. Remaining joints in the lining are not attached to the vessel. The lining is further supported by pressure-bulging the lining over a pattern of 2-in. diam. × 1/4-in. thick steel buttons welded to the shell at 2-ft intervals, and also by maintaining a vacuum between the lining and the shell. Under variations of pressure and temperature, an austenitic lining may expand or contract radially and axially. When temperature and pressure are high, the lining is in tight contact with the shell and in a state of compression. While the shell remains hot, the lining may be cooled by filling with cold chips. Axial tension develops by reason of retention by the buttons and by the girth attachment joints. In addition, the lining is free to contract radially, without stress except at the girth attachment joints and nozzles, where bending produces added tension stresses equal to 182% of the hoop stresses at the weld. The radial and axial stresses combined produce stresses of the order of 60,000 p.s.i. with a possibility of subsequent fatigue failure. Reducing the stress at points of fixity is made possible by the use of transition members which reduces direct stress and minimizes bending by avoidance of concentration of bending strain.

THE KOPPARFORS bag lining is a patented method of protection of the inner surfaces of vessels, which may be required to contain liquids, gases, or solids corrosive or abrasive to carbon steel (*1*). The lining is fabricated from suitable corrosion-resistant materials which may be installed in new vessels or in older vessels which have been partially corroded in service.

The lining is applied, principally, in the form of large sheets welded to one another and to nozzle liners to form a continuous vaportight shield in the vessel. Although the bag is supported at each of the nozzle attachments, the principal supports are two or more circumferential joints attached to the mild steel shell, by a grid of buttons welded to the inner surface of the shell prior to installation of the lining and, when in service, by a vacuum maintained between vessel and lining. This vacuum is an important feature of the patent and has a major effect on the structural stability of the lining.

The linings are thin and are not considered to share in the structural or pressure strength of the vessel. Nevertheless, they must be pressure tight to contain liquids or gases, even if they are subjected to greater stress than the body of the vessel. Such greater stresses may be due to installation techniques, thermal cycling, the use of corrosion-resistant materials which differ from carbon steel in thermal expansion or contraction, and to the methods followed in operation of the vessel.

If the number of cyclic changes in temperature and pressure for the expected life of a vessel is large, a possibility exists of fatigue failure in the lining.

FORMER LININGS

Because of these factors it was customary to line vessels with suitable lining metals, which are welded to the vessel with welds at closely spaced intervals. This is commonly known as "strip lining" where the width of strip is usually restricted to 6 in. or less. The width of these strips usually decreases in proportion to increasing temperature ranges. If the spacing between welds is too great, fatigue stresses may induce failure in the welds or lining metal. Large thin sheets have been used for lining purposes, but attachment welds in the form of plug, fillet, butt, slot or resistance welds are usually closely spaced over the area of the sheets to avoid overstressing of the liner. However, each weld presents a possibility of leakage and a focal spot for fatigue and for corrosion because of the difficulty in obtaining suitable chemistry in the weld metal. It is therefore highly desirable to use the least amount of weld consistent with good design and practice.

The Kopparfors bag lining has accomplished this weld reduction by using large sheets of lining metals which have a minimum of attachment to the vessel. It is important, however, to know and provide for the stress conditions existing by reason of installation practices, thermal cycling, and restraint at points of attachment. The following analysis will establish these stress conditions and enable a proper assessment to be made of the structural strength of the lining and its details of design.

STRAIN AND STRESS CONDITIONS

When a Kopparfors lining is being installed in a welded vessel, 2-in. diameter buttons, 1/4-in. thick, are welded to the inside of the shell at 2-ft. intervals. The lining is then installed inside of the buttons, 1/4 in. clear off the shell. Welds between sheets and to the vessel at the points of attachment must be at least as strong as the lining material since, after installation, hydraulic pressure is applied inside the lining to stress the shell to 50% above the design stress of the vessel. This load will produce plastic flow of the lining by bulging it around the buttons and against the shell. After release of the hydraulic pressure, both shell and lining will recover elastically, leaving a small gap between. This gap, in practice, is unavoidable. It is important in the stress analysis because it permits bending stresses to occur in the lining at the points of attachment to the vessel, and causes a redistribution of stresses arising from pressure and temperature.

G. CAPE, and A. M. BAIN, Dominion Bridge Co., Ltd., Montreal, Quebec, Canada

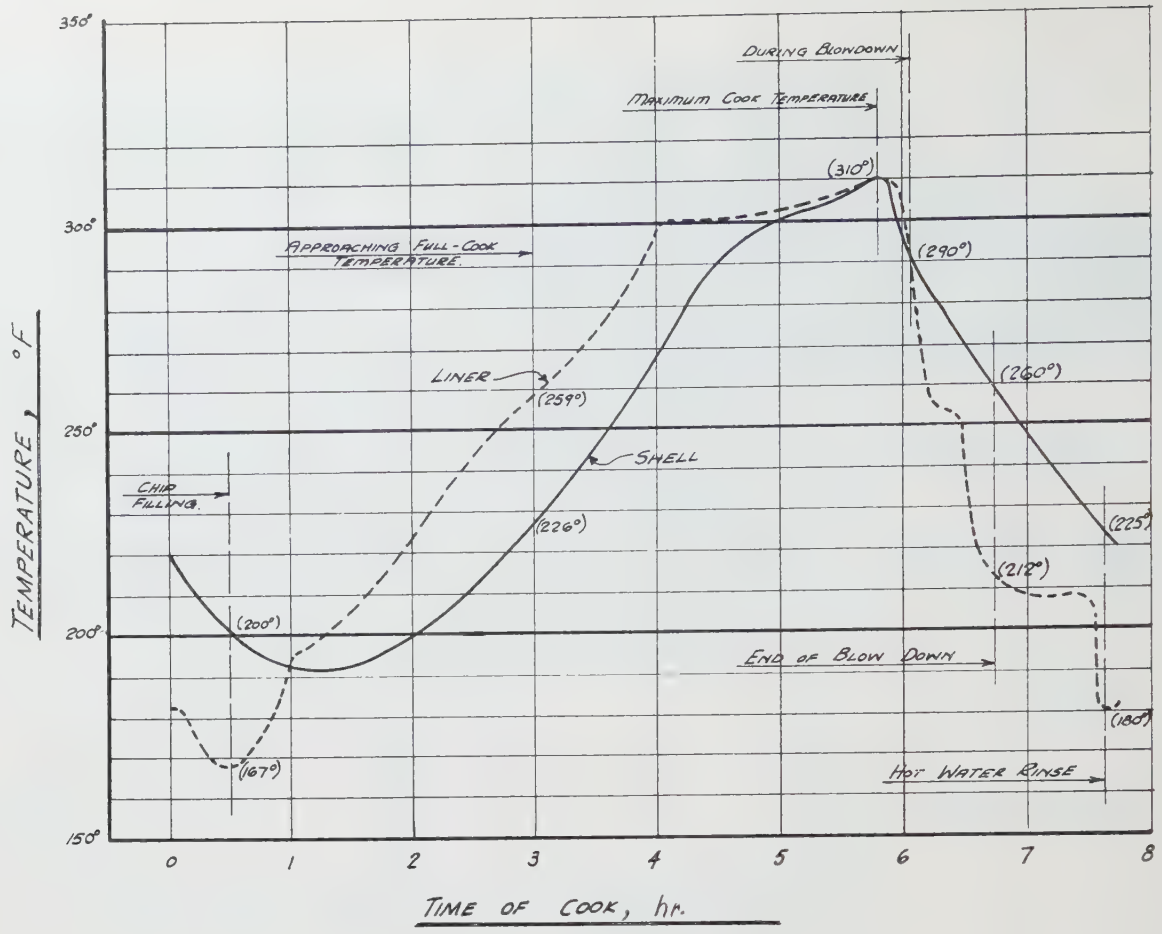

Fig. 1. Temperature cycle; stainless clad sulfite digester

When the vessel is later placed in service, variable stress conditions will be found in the lining because of pressure and temperature changes.

Pressure acting on the liner will expand it radially to first close the gap between liner and shell, and then expand both liner and shell. Greater hoop stresses will be produced in the liner. In the axial direction, strain in liner and shell will be the same.

Temperature acting on the liner will strain it both radially and axially. Strain will be greater if the liner is made of austenitic stainless steel, which has a coefficient of thermal expansion about 50% more than carbon steel. Axially, strain will be resisted by the attachment joints to the vessel. Tension or compression stresses will result. Buckling of the thin liner due to compressive strains would be avoided by reason of the support from the shell and the vacuum holding the liner against the vessel.

Radially, expansion of the liner is restrained at the attachment joints, but is free elsewhere because of the installation gap between the liner and the vessel. Flexing of the liner in the radial direction causes longitudinal bending stresses at the points of restraint. By comparison with the stiffened pipe theory, these stresses are 182% of the corresponding hoop stresses. Furthermore, bending stresses are additive with axial stresses. The combination can produce maximum stresses exceeding the yield strength for each cyclic change, and a possibility of fatigue failure can exist unless the structural details are arranged to prevent it.

A digester is subject to a considerable variation in temperature and pressure during any cooking cycle. To establish authentic working data, a chart of simultaneous temperatures was obtained for the inside and outside surfaces of a clad and insulated sulfite digester (see Fig. 1). Table II gives these temperatures and their corresponding pressures. The dimensional data for the same digester are shown in Table I, and have been used in the subsequent calculations.

Table I. Vessel Data

Design pressure including static head P, p.s.i.	148
Static head at lower knuckle, p.s.i.	13
Vessel thickness (t_v), in.	1.75
Vessel radius (R), in.	117
Lining material	317L Stainless steel
Shell material	Carbon steel

Table II. Operating Conditions

	Temperature, °F. Lining	Shell	Gage press., p.s.i.g.	Static head p.s.i.	Working press., p.s.i.
1. Approaching cooking temp.	259	226	27	13	40[a]
2. Max. cooking temperature	310	310	90	13	103
3. During blowdown	290	290	70	13	83[a]
4. End of blowdown	212	260	0	0	0
5. Hot-water rinse	180	225	0	0	0
6. Chip filling (normal)	167	200	0	0	0
7. Assumed cold chip fill	65	190	0	0	0

[a] Estimated pressures.

The lining will be appreciably affected by temperatures and pressures similar to those in Table II. Stresses and strains will be produced. They will occur in two

Table III. Basic Constants

Y.P. of lining—Type 317L stainless, p.s.i.	40×10^3 [a]
Thickness of lining (t_L), in.	0.125
Young's modulus of elasticity (E), p.s.i.	30×10^6
Strain at Y.P., (lining), in./in.	1.33×10^{-3}
Linear coeff. of expn. of mild steel (K_v), in./in./°F	6.5×10^{-6}
Linear coeff. of expn. of stainless steel (K_L), in./in.°F	9.2×10^{-6}

[a] Estimated yield point of material after some cold work.

principal directions, radially and axially. Since we can convert unit radial strains directly into either radial movements or hoop stresses, and since axial strains can be converted directly into axial stresses, these extremely small units have been used throughout the subsequent calculations and tables.

Physical Constants

The basic constants used in the computations are shown in Table III.

It was assumed that the lining would be fabricated from 317L material, $^1/_8$-in. thick, in the annealed condition, rolled to radius, and installed in large sheets in contact with the $^1/_4$-in. steel buttons. The joints would be tacked together and later welded, leaving a gap of $^1/_{16}$ in. clear off the buttons. Welds between sheets or attachment welds would need to be at least as strong as the lining sheet. Attachment to the shell would be made only at top and bottom of the cylindrical shell portion of the vessel. Nozzle attachment welds need not be considered at this time. It was also assumed that installation temperature would be 90°F., if carried out in a warm digester room.

Pressure Testing. After completion of welding, the lining and vessel would be subjected to a hydrostatic pressure test 50% above the design pressure of the vessel. The temperature of the water was assumed to be 65°F.

Calculations of vessel and lining strains during and after pressure test were then made as follows:

Button thickness, in.	0.25
Installation gap—lining-to-buttons, in.	0.063
Total gap between shell and lining, in.	0.321
Thermal contraction—lining (90° − 65°) RK_L, in.	0.027
Thermal contraction—vessel (90° − 65°) RK_v, in.	0.019
Difference in contraction—lining and vessel, in.	0.008
Unit strain in lining before contacting shell = 0.321/117, in./in.	0.00275
Combined lining and shell unit strain = $\frac{148 \times 1.5 \times 117}{(1.75 + 0.125)E}$ in., in.	0.00046
Total unit strain in lining, in./in.	0.00321

From tables of stress–strain relationship, the strain of 0.00321 in per in. (0.32%) would cause plastic flow and dimpling of the lining around the buttons and against the shell. This strain would represent a stress of not more than 40,000 p.s.i. in the nondimpled portions of the lining.* At completion of testing, when dropping the pressure to atmospheric, the shell and lining would both contract, ending with the following radial clearances in inches.

Relaxation of lining, $40{,}000 \times 117/E$	0.1560
Relaxation of shell, 0.00046×117	0.0540
Therefore, gap between lining and shell after test pressure at 65°F	0.102
Reduction of gap on warming to 90°F. = $(K_L - K_v) \times 117 \times (90° - 65°)$	0.008
Therefore, gap between lining and shell at 90°	0.094

Vacuum Effect. The lining is, however, subject to another force which tends to reduce the gap existing after plastic-molding the lining inside of the digester by pressure-testing. This force is due to the maintenance of a vacuum between lining and shell during all phases of the digester cooking cycle. A water-jet pump can maintain a vacuum equal to 90% of atmospheric pressure, i.e., $0.9 \times 14.7 = 13.2$ p.s.i. This has the same effect on the lining as an internal pressure.

The radial movement of the lining due to vacuum will then be:

$$PR^2/t_L E = \frac{13.2 \times 117^2}{^1/_8 \times 30 \times 10^6} = 0.048 \text{ in.}$$

Therefore, the gap between lining and shell under vacuum, at digester room temperature of 90°F. is $0.094 - 0.048 = 0.046$ in.

These figures give the dimensions of the installed lining ready for service. It is now possible to analyze what happens during the various stages of the cooking cycle. It is convenient, however, to determine first some of the factors which will appear in the later equations. These are as follows:

Effect of Variables

1. Pressure inside of the vessel will force the lining into contact with the shell and the two materials will, from the time of contact on, jointly resist the load. The strain in the vessel produced by pressure may then be calculated from the formula:
Unit strain $= PR/TE = PK_p$

where $K_p = \frac{R}{TE} = \frac{R}{(t_V + t_L)E} = \frac{117}{(1.75 + 0.125)\,30 \times 10^6} = 2.08 \times 10^{-6}$ in./in.

2. The stress in the lining produced by vacuum only will be:

$$\frac{PR}{t_L} = \frac{13.2 \times 117}{0.125} = 12{,}400 \text{ p.s.i.}$$

This stress is one of the stresses shown in Tables IV and V.

3. Unit strain in lining by vacuum $= 12400/E = 4.13 \times 10^{-4}$ in./in.

4. Unit strain in shell by vacuum $= \frac{13.2 \times 117}{1.75E} = 2.9 \times 10^{-5}$ in./in.

5. Temperature will strain both lining and shell separately in accordance with the formula:

Unit strain $= TK_v$ for mild steel vessel
or TK_L for stainless steel lining

6. The gap of 0.094 in. between lining and shell after pressure testing is equivalent to a unit strain of $0.094/117 = 8.04 \times 10^{-4}$ in per in. This gap is more than the strain induced by the vacuum only, which was 4.8×10^{-4} in. per in. This means that the lining, with vacuum on, will take the full vacuum load without bearing against the shell.

* Nickel-alloy steels—section 7, International Nickel Co., Inc., N. Y.

Table IV. Constants for the Seven Operating Conditions in Table II

	Function		Unit strain (in./in.) or stress (p.s.i.) or gap (in.) or press. (p.s.i.) — Radial: Fixing joint	Radial: Free areas	Axial: All areas
1.	Strain in V[a] by temp., (226 − 90) K_v	a	0.000883	0.000883	0.000883
	Strain gap between V and L,[b] installation	b	0	0.000804	0
	Strain sum, $a + b$	V	0.000883	0.001687	0.000883
	Strain in L by temp., (259 − 90)K_L	L	0.001556	0.001556	0.001556
	Strain difference, V − L	c	−0.000673	+0.000131	−0.000673
	Pressure on L to close gap, $c\ K_g$			4.2	
	Strain in V by press, 40 K_p	d	+0.000083		+0.000041
	Strain in V by press, (40 − 4.2)K_p	d		0.000074	
	Strain in L, $c + d$		−0.000590	+0.000205	−0.000632
	Stress in L		17,770C[c]	6,150T[d]	18,960C
	Final gap		0	0	0
2.	Strain in V by temp., (310 − 90)K_v	a	0.001430	0.001430	0.001430
	Strain gap between V and L, installation	b	0	0.000804	0
	Strain sum, $a + b$	V	0.001430	0.002234	0.001430
	Strain in L by temp., (310 − 90)K_L	L	0.002025	0.002025	0.002025
	Strain difference, V − L	c	−0.000595	+0.000209	−0.000595
	Pressure on L to close gap, $c\ K_g$			6.7	
	Strain in V by press., 103 K_p	d	0.000214		0.000107
	Strain in V by press., (103 − 6.7)K_p	d		0.000201	
	Strain in L, $c + d$		−0.000381	+0.000410	−0.000488
	Stress in L		11,430C	12,340T	14,640C
	Final gap		0	0	0
3.	Strain in V by temp., (290 − 90)K_v	a	0.001300	0.001300	0.001300
	Strain gap between V and L, installation	b	0	0.000804	0
	Strain sum, $a + b$	V	0.001300	0.002104	0.001300
	Strain in L by temp., (290 − 90)K_L	L	0.001840	0.001840	0.001840
	Strain difference, V − L	c	−0.000540	+0.000264	−0.000540
	Pressure on L to close gap, $c\ K_g$			8.4	
	Strain in V by press, 83 K_p	d	0.000173		0.000086
	Strain in V by press, (83 − 8.4)K_p	d		0.000155	
	Strain in L, $c + d$		−0.000367	+0.000419	−0.000454
	Stress in L		11,010C	12,570T	13,620C
	Final gap		0	0	0
4.	Strain in V by temp. (260 − 90)K_v	a	0.001105	0.001105	0.001105
	Strain gap between V and L, installation	b	0	0.000804	0
	Strain sum, $a + b$	V	0.001105	0.001909	0.001105
	Strain in L by temp., (212 − 90)K_L	L	0.001121	0.001121	0.001121
	Strain difference, V − L	c	−0.000016	+0.000788	−0.000016
	Strain due to vacuum on L	d	0	0.000413	0
	Strain due to vacuum on V	e		0.000029	
	Strain net, $c - d - e$	f	−0.000016	+0.000346	−0.000016
	Stress		480C	12,400T	480C
	Final gap, fR			0.040	
5.	Strain in V by temp. (225 − 90)K_v	a	0.000877	0.000877	0.000877
	Strain gap between V and L, installation	b	0	0.000804	0
	Strain sum, $a + b$	V	0.000877	0.001681	0.000877
	Strain in L by temp., (180 − 90)K_L	L	0.000828	0.000828	0.000828
	Strain difference, V − L	c	0.000049	0.000853	0.000049
	Strain due to vacuum on L	d	0	0.000413	0
	Strain due to vacuum on V	e	0	0.000029	0
	Strain net, $c - d - e$	f	0.000049	0.000411	0.000049
	Stress		1,470T	12,400T	1,470T
	Final gap, fR			0.048	
6.	Strain in V by temp., (200 − 90)K_v	a	0.000715	0.000715	0.000715
	Strain gap between V and L, installation	b	0	0.000804	0
	Strain sum, $a + b$	V	0.000715	0.001519	0.000715
	Strain in L by temp. (167 − 90)K_L	L	0.000709	0.000709	0.000709
	Strain difference, V − L	c	0.000006	0.000810	0.000006
	Pressure		0	0	0
	Strain due to vacuum on L	d	0	0.000413	0
	Strain due to vacuum on V	e	0	0.000029	0
	Strain net, $c - d - e$		0.000006	0.000368	0.000006
	Stress		180T	12,400T	180T
	Final gap			0.043	
7.	Strain in V by temp. (190 − 90)K_v	a	0.000650	0.000650	0.000650
	Strain gap between V and L, installation	b	0	0.000804	0
	Strain sum, $a + b$	V	0.000650	0.001454	0.000650
	Strain in L by temp., (65 − 90)K_L	L	0.000230	0.000230	0.000230
	Strain difference, V − L	c	0.000880	0.001680	0.000880
	Pressure		0	0	0

Function		Unit strain (in./in.) or stress (p.s.i.) or gap (in.) or pressure (p.s.i.) Radial: Fixing joint	Radial: Free areas	Axial: All areas
Strain due to vacuum on L	d	0	0.000413	0
Strain due to vacuum on V	e	0	0.000029	0
Strain net, $c - d - e$		0.000880	0.001238	0.000880
Stress		26,400T	12,400T	26,400T
Final gap			0.145	

[a] Vessel; [b] Lining; [c] Compression; [d] Tension.

7. When a difference of unit strain exists between liner and shell because of temperature and the original installation gap, a small pressure applied inside the vessel will further strain the lining into initial contact with the shell.

The pressure on the lining to close the gap (unit strain) is given by:

$$\frac{ft}{R} = \text{Unit strain} \times E \times \frac{T}{R}$$
$$= \text{Unit strain} \times K_g$$

where

$$K_g = \frac{ET}{R} = 32{,}000 \text{ p.s.i.}$$

Radial and Axial Strains. As stated previously, the lining will be appreciably affected by the temperatures and pressures listed in Table II. Strains will be produced in radial and axial directions.

Radially, it will be necessary to consider the strains in two different areas; i.e., those areas immediately adjacent to welds attaching the lining to the vessel termed "fixing joint," and those areas which are remote from the immediate area of attachment and which are consequently not influenced by the rigidity of the vessel wall. These are termed "free areas." Axially, since the lining is attached at top and bottom of the shell course, tension or compression stresses and strains will occur uniformly over all areas.

Strains in the lining in the two principal directions of stress and in the two areas of restraint and freedom, can now be calculated using the several operating conditions in Table II, by comparing the difference in strain of shell and lining separately in each case. The results are shown in Table IV and have been listed in inches per inch for easy conversion into the unit stresses of Table V.

The data in Table IV include computations for the gap existing between liner and shell while under a vacuum, at various stages of the digester cook. The maximum gap appears to be 0.048 in. during water wash. It might be thought then, that this gap would result in the lining being free of the restraining buttons and thus render them ineffective. However, during the time required for chip filling, the digester shell would be further cooled and the gap would decrease. Even if the lining is cooled and shrinks near the bottom by chip addition, at any elevation above the chip level, the lining is warm, expanded, and therefore effectively keyed to the buttons to prevent its downward sag. Then with liquor addition, the hydrostatic head and liquor temperatures tend to force the lining below the chip level against the buttons. Thus, it would appear that the lining is keyed to the buttons both above and below the ascending level of the chip bed to reduce shear strain elsewhere, particularly around nozzle connections.

Since the potential strain differences determined in Table IV cannot in specific cases occur because of the attachment and contact of lining with shell, they have been used to calculate the corresponding stresses which must exist to prevent them. These stresses are calculated from the formula: Stress = $E \times$ Strain, and are summarized in Table V.

Table V. Stresses in Lining from Table IV Strains

Operating condition	Stress, p.s.i. Hoop (radial): Fixing joint	Hoop (radial): Free areas	Axial, All areas
1. Approaching cooking temperature	17,770C	6,150T	18,960C
2. Max. cooking temperature	11,430C	12,340T	14,640C
3. During blowdown	11,010C	12,570T	13,620C
4. End of blowdown	480C	12,400T	480C
5. Hot-water rinse	1,470T	12,400T	1,470T
6. Normal chip filling	180T	12,400T	180T
7. Assumed cold chip filling	26,400T	12,400T	26,400T

A study of Table V shows that hoop stresses, calculated from radial strains in the lining at the fixing joint and at the free areas, vary in amount and in sign (compression or tension). Thus for operating conditions no. 1, the lining at the fixing joint is in compression. In effect, the lining at this point is moved inwardly relative to the free areas. Adjacent free areas, however, expand outwardly. Longitudinal bending of the liner at the fixing joint, consequently, occurs in direct proportion to the sum of the two separate hoop stresses; i.e., bending stresses are proportional to a combined stress of 17,700 + 6,150 = 23,920 p.s.i.

This same figure can, for operating conditions nos. 1, 2, and 3, be arrived at more simply by calculating the amount of hoop stress needed in the lining to force it into contact with the shell. For conditions nos. 4 to 7, however, the method of combining free and fixed area hoop stresses must be used. Operating conditions nos. 2, 3, and 4 likewise produce similar additive hoop stresses. Operating conditions nos. 5, 6 and 7, however, produce bending conditions which are proportional to the difference in value of the hoop stresses. This is because at the fixing joint, the lining is in effect pulled outwardly while adjacent free areas move in the same direction to relieve the bending.

The hoop stresses, when combined in this manner, will produce longitudinal bending stresses if the liner is locally restrained by any ring-attachment joint and the remainder is free to move.

Bending Stresses in Lining

The extent to which bending stresses can occur in a case of this kind has been demonstrated by Schorer(*2*). Schorer's analysis was made of a penstock which had

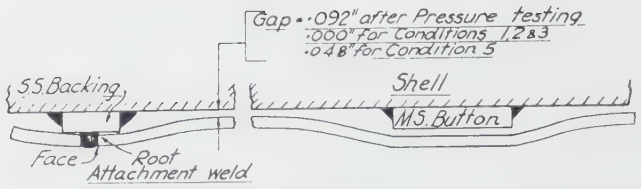

Fig. 2. ***Gap between bag liner and shell at different stages of cooling cycle***

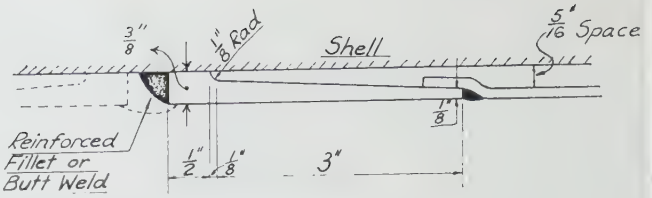

Fig. 3. ***Transition member and liner position before pressure test***

rigid circular girders encircling the penstock at remote distances from each other. He showed that longitudinal bending stresses in the shell, at the restraining girders, are 182% of the hoop stresses induced by pressure in the penstock. Since the analogy of flexible penstock shells and rigid circular girders applies to the case of a thin bag lining attached to a rigid vessel by widely spaced girth attachment welds, the same ratios of hoop and bending stresses apply. Therefore it follows that the actual stresses in bending of the lining at the point of attachment to the shell will be 1.82 times the hoop stresses, if the lining is not in contact with the shell between points of attachment.

Bending stresses produce tension and compression forces which are equal to one another on the outer and inner faces of the lining, at the points of attachment to the shell. Also, because bending stresses are parallel to the axis of the vessel, the axial stresses become additive with the bending stresses. For this reason, it is necessary to determine the amount and kind of stress existing on each face of the lining; i.e., at the inner face of the lining, next to the liquor, and at the root of the attachment weld (see Fig. 2 and Table VI).

Stress Intensifier. Table VI shows that for the first six operating conditions the inner face of the lining is subject to stresses varying from 21,370 to 29,180 p.s.i. tension, while the root of the attachment weld is stressed to 62,460 p.s.i. compression with a swing of 44,000 p.s.i. The stresses on the inner face are only moderately high, but stresses at the root are beyond the yield point of the stainless steel, particularly in view of the notch created at the junction of the shell and lining in the weld metal. Such a notch could produce an intensification of stress to cause yield in compression, which would swing to tension on decrease of compression loading. Fatigue-cracking then could occur. Indeed, experience in Sweden with earlier digesters, showed that attachments of the liner to the shell, as in Fig. 2, were prone to failure after comparatively short periods of use. Various methods were tried to overcome this difficulty, including the use of annular buckles close to the attachment welds to decrease the axial stresses. The buckles did not, however, alleviate the hoop strains with their concurrent high bending stresses, and other means were devised.

Effect of Rapid Cooling. The last operating condition, no 7, shown in Table VI, has been included as an indication of the stress condition which could arise if the lining were cooled by means of cold-water washing or cold-chip filling. Such drastic cooling is not advisable in view of the high stresses produced. Fatigue failure is very probable.

Transition Member. The foregoing calculations led to a consideration of a thickened transition piece of stainless steel, which would be anchored to the vessel with substantial weld to decrease the notch stress intensity, and which would taper to the liner thickness in a reasonable distance to reduce the bending moment. A transition member such as shown in Fig. 3 resulted. It was then considered expedient to determine the stress conditions existing in such a transition member when anchored to the shell and welded to the lining. A further study was then made, applying the results of Schorer's analysis to this variable thickness member. The Dominion Bridge Co. has applied for patents on the transition member in Canada, the U.S.A. and Sweden (*3*).

Analogy With Stiffening Tube. Figure 4 illustrates Schorer's analysis of bending in large diameter pipes in the vicinity of restraining rings. The formulas, which are computed in Schorer's work, take into account the flexibility of the restraining ring. The formula in Fig. 4, for bending stress and also for H, the shear at the restraining ring, are derived from Schorer on the assumption that the restraining ring is inflexible. This is quite reasonable since, in bag linings, the Schorer restraining ring is the relatively thick and inflexible wall of the vessel and Schorer's pipe is the thin flexible liner.

Temperature Effects. Schorer's formulas are based on internal pressure in a large pipe. In bag linings, both pressure and temperature strain the lining. There is no difficulty in determining the bending stresses in bulging the lining against the shell by reason of pressure. Temperature effects cannot be directly computed, and it becomes essential, therefore, to

Table VI. Maximum Stress (p.s.i.) in Lining at Fixing Joint

Location	*Stress*	*Operating condition* 1	2	3	4	5	6	7
Face	Combined hoop	23,920	23,770	23,580	12,880	10,930	12,220	14,000
	C.H × 1.82	43,500T	43,200T	42,800T	23,500T	19,900T	22,200T	25,500C
	Axial	18,960C	14,640C	13,620C	480C	1,470T	180T	26,400T
	Total	24,540T	28,560T	29,180T	23,020T	21,370T	22,380T	900T
Root	C.H. × 1.82	43,500C	43,200C	42,800C	23,500C	19,900C	22,200	25,500T
	Axial	18,960C	14,640C	13,620C	480C	1,470C	180T	26,400T
	Total	62,460C	57,840C	56,420C	23,980C	18,430C	22,020C	51,900T

establish an analogy whereby the bending stresses caused by temperature can be determined.

Figure 5 shows the deflection occuring by reason of temperature in the portion of liner adjacent to the restraining point.

Analogy with Variably Loaded Beams. Since Schorer's analysis was computed on the basis of pressure loading, it becomes essential in applying his findings to convert temperature-deflection effects into assumed load effects.

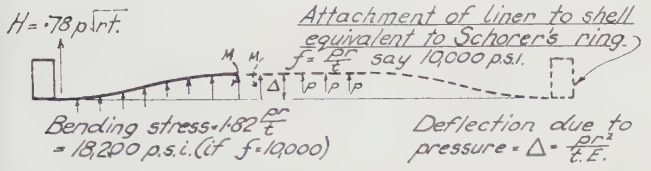
H = ·78 p √rt.
Attachment of liner to shell equivalent to Schorer's ring.
f = pr/t say 10,000 p.s.i.
Bending stress = 1.82 pr/t = 18,200 p.s.i. (if f = 10,000)
Deflection due to pressure = Δ = pr²/t.E.

Fig. 4. Stress and deflection of ring-reinforced pipe [Schorer's analysis (2)]

Notation

H = shear at restrained end, lb.
b = width of beam (1 in. throughout)
Δ = deflection, in.
f = hoop stress, p.s.i.
L = length of beam, in.
p = load (p.s.i.) produced by pressure, or load produced by temperature causing equivalent pressure strain
r = inside radius of vessel, in.
t = thickness of liner, in.

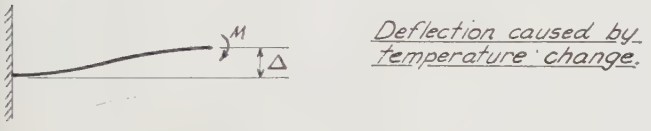

Fig. 5. Representation of deflection in restrained cylinder due to temperature change

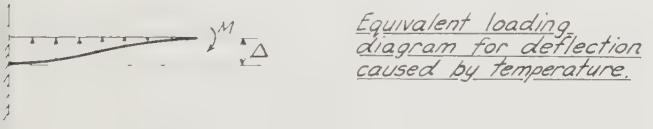

Fig. 6. Pressure loading corresponding to temperature deflection

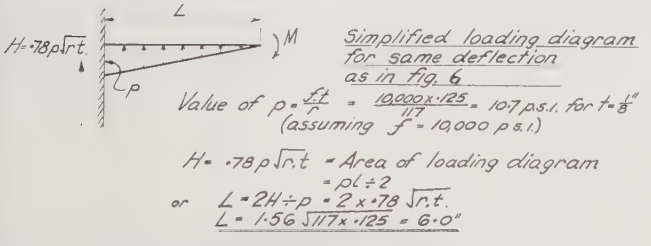
H = ·78 p √rt.
L
M
p
Simplified loading diagram for same deflection as in fig. 6
Value of p = ft/r = 10,000 × ·125 / 117 = 10.7 p.s.i. for t = 1/8"
(assuming f = 10,000 p.s.i.)
H = ·78 p √r.t = Area of loading diagram = pL ÷ 2
or L = 2H ÷ p = 2 × ·78 √r.t.
L = 1·56 √117 × ·125 = 6·0"

Fig. 7. Simplified loading corresponding to temperature deflection

Notation

Beam—One end fixed, other end free vertically but guided with restraining external moment. Uniform depth (1/8 in.) throughout its length.

H = shear at restrained end, lb.
b = width of beam (1 in. throughout)
f = hoop stress, p.s.i.
L = length of beam, in.
p = load (p.s.i.) produced by pressure, or load produced by temperature causing equivalent pressure strain
r = inside radius of vessel, in.
t = thickness of liner, in.

Figure 6 shows the necessary assumed loading on a cantilever beam with a guided outer end, parallel end tangents and an end moment. These conditions are similar to those of Fig. 4, where the portion of the penstock adjacent to the restraining ring is shown.

Figure 7, represents, for simpler calculations, an approximately equivalent loading, based on a straight

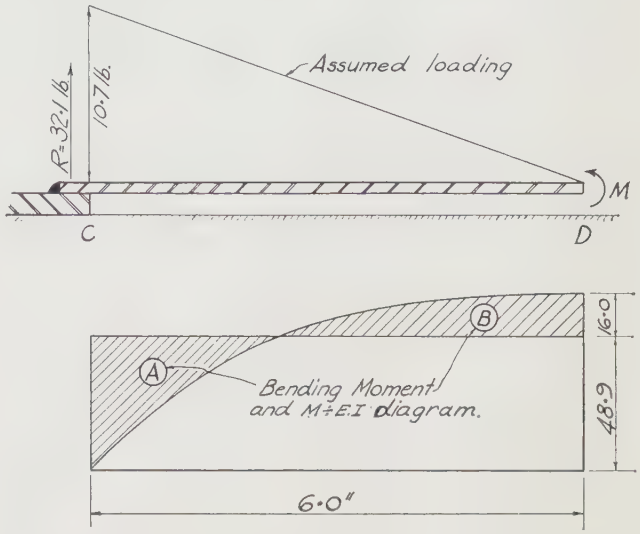

Fig. 8. Assumed loading required on liner adjacent to fixed point to produce known deflection

For zero slope at C and D area A = area B. Δ = deflection at D is equal to the algebraic sum of the moments of Areas A and B taken about D and divided by EI

$$\Delta(\text{due to } f = 10{,}000 \text{ p.s.i.}) = \frac{10{,}000 \times 117}{30{,}000{,}000}$$

$$\Delta = 0.036 \text{ and } 0.039 \text{ in.}$$

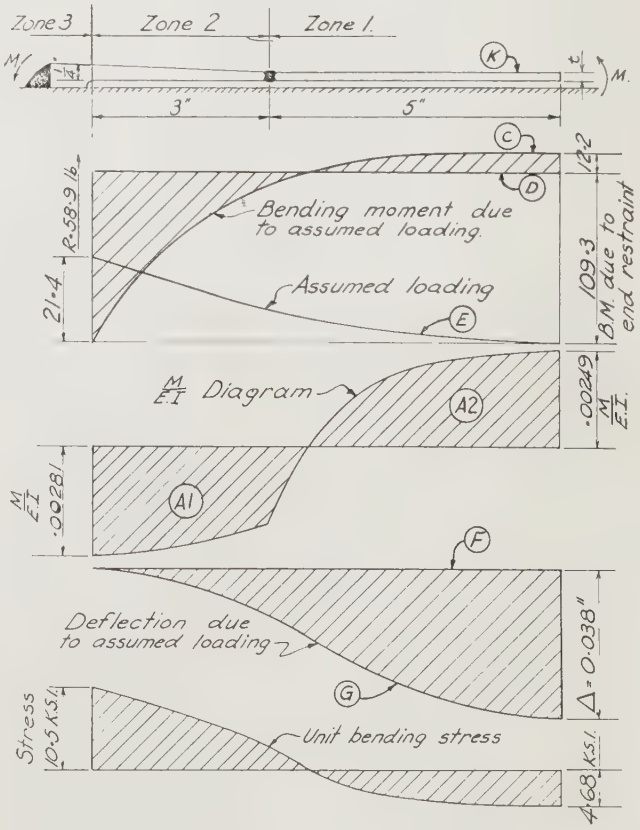

Fig. 9. Transition member bending stresses.

Table VII. Maximum Stresses (p.s.i.) in Transition Member at Junction of Zones *2* and *3* of Fig. 9

Location	Stress	Operating condition 1	2	3	4	5	6	7
Face	Combined hoop	23,920	23,770	23,580	12,880	10,930	12,220	14,000
	C.H. × 1.05	25,100T	25,000T	24,700T	13,500T	11,480T	12,800T	14,700C
	Axial	9,480C	7,320C	6,810C	240C	730T	90T	13,200T
	Total	15,620T	17,680T	17,890T	13,260T	12,210T	12,890T	1,500C
Root	C.H. × 1.05	25,100C	25,000C	24,700C	13,500C	11,480	12,800C	14,700T
	Axial	9,480C	7,320C	6,810C	240C	730T	90T	13,200T
	Total	34,580C	32,320C	31,510C	13,740C	10,750C	12,710C	27,900T

line load variation instead of the curve of Fig. 6. The value of p needed for a hoop stress of say, 10,000 p.s.i. in a cylindrical liner $^1/_8$ in. thick and 117 in. radius is found to be 10.7 p.s.i. The length of the cantilever beam with parallel end tangents (zero slope) is then found to equal 6 in.

Figure 8 shows that the deflection of 0.036 in. in the beam of Fig. 7, is sufficiently close to the deflection of a cylinder under pressure, 0.039 in., to prove the correctness of the assumed loading.

Figure 9 shows a view of a cantilever beam (K) with guided end and end moment (as in Fig. 6), in which hand end (the center of the double restrained end beam) will then be equal to the deflections of the beam. These deflections are shown as curve G. The loading which corresponds to these deflections, when compared with the initially assumed loading, is found to be in close agreement. Therefore, recalculations of bending moments, etc., for a closer approximation is not needed.

The maximum deflection of the beam, as calculated, is 0.038 in. The maximum deflection of the liner under the initially assumed hoop stress of 10,000 p.s.i. = $10{,}000/E \times$ radius of liner = 0.039 in. This checks the accuracy of the assumptions closely.

Table VIII. Maximum Stresses (p.s.i.) in Transition Member at Attachment Weld

Location	Stress	Operating condition 1	2	3	4	5	6	7
Face	Bending	11,110T	11,100T	10,950T	5990T	5100T	5680T	6,520C
	Axial	6,300C	4,900C	4,500C	160C	500T	60T	8,800T
	Total	4,810T	6,200T	6,450T	5830T	5600T	5740T	2,280T
Root	Bending	11,110C	11,100C	10,950C	5990C	5100C	5680C	6,520T
	Axial	6,300C	4,900C	4,500C	160C	500T	60T	8,800T
	Total	17,410C	16,000C	15,450C	6150C	4600C	5620C	15,320T

a transition member is interposed between the restrained end connection and the liner. The shape of the transition member is designed to reduce combined stresses and is varied in thickness in the three zones marked *1*, *2*, and *3*.

Zone *1*, represents the liner proper. Zone *2* represents a portion of the transition member in which the thickness gradually increases as bending moment increases. Zone *3*, represents a still greater increase in thickness so that the cross section at the attachment weld is sufficiently great to absorb the concentrated bending stresses at the notch formed at the root of the attachment weld.

Figure 9 also shows the following. Line F represents the beam composed of transition member (zone *2*) and liner (zone *1*). Line E represents the assumed loading, similar to Fig. 7 but having a p value at the left of 21.4 for $^1/_4$-in. thickness (or 2 × 10.7), and varying to zero at the right. Line C represents the bending moment due to assumed load. Line D representing the restraining moment whose value of 109.3 lb. is so established that in the M/EI diagram, area $A1 = A2$. When these areas are equal, the slope at each end of the cantilever beam with guided end is equal to zero. This is the same as half a beam of twice the length restrained at both ends and deflected symmetrically so that the slope at its center is zero.

Moments of the M/EI diagram taken about the right-

The information on Fig. 9 now permits us to use the bending moment diagram with confidence. Since $f = M/S$, then at the extreme left, where $t = {}^1/_4$ in., $f = 109.3 \times 6 \times 16 = 10{,}500$ p.s.i.

Since the hoop stress is 10,000 p.s.i., and the corresponding bending stress in the transition member is a maximum of 10,500 p.s.i., the fixing factor for bending is then 105% of the hoop stress. This shows the value of the transition member since direct attachment of the thin liner to the shell would result in a bending stress equal to 182% of the hoop stress (*2*). The factor of 105% instead of 182%, applied to the stresses of Table VI, would obviously increase the fatigue life appreciably.

Effect of Transition Member

Table VII may then be derived from Table VI by changing the bending factor from 1.82 to 1.05 and by reducing the axial stress by half. The latter reduction is due to the fact that the axial load in the liner is now carried by the transition member, which is twice as thick at the point considered.

It would be feasible to increase the thickness and length of the transition member slightly to reduce further the stresses of Table VII to lower values. However, the maximum stress of 34,580 p.s.i. compression is well below the yield point, and the maximum swing of 34,580 − 10,750 = 23,830 p.s.i., is well below the endurance limit of the metal (see Fig. 10).

It is believed these values are quite reasonable, particularly in view of the fact that there is very little notch effect at the junction of the $^3/_8$-in. and $^1/_4$-in. portions of the transition member. The attachment weld at the end of the $^3/_8$-in. thick portion does, however, have a notch with a consequent unknown concentration of stress. The stress at this point, however, is considerably lower by reason of the greater thickness which reduces both bending and axial stresses. Table VIII shows these stresses.

Tables VII and VIII show that under operating conditions nos. 1 to 6 the stress on the face, or liquor side, of the transition member will not exceed 17,890 p.s.i. tension, and the maximum variation only 5680 p.s.i. The stress on the root side during the same normal cooking cycle reaches 34,580 p.s.i. compression, and the total variation at the same point is only 17,830 p.s.i. All these stresses are well within the endurance limit of the material.

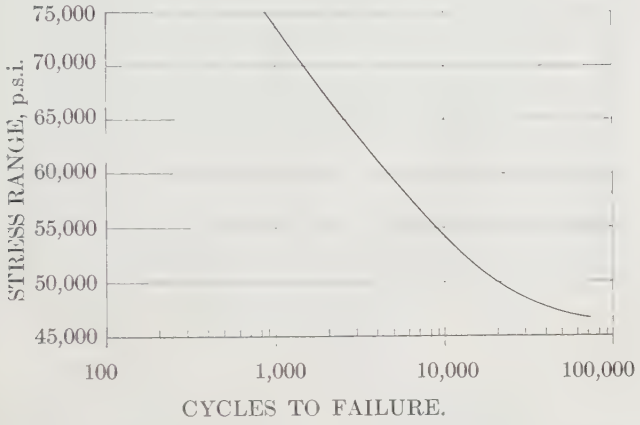

Fig. 10. Stress range vs. cycles-to-failure under thermal cycling conditions, austenitic stainless steel

If we consider the case of undesirable chilling of the liner, as in operating condition no. 7, we get a total stress variation from Table VII of 62,480 p.s.i. with no notch effect involved, and from Table VIII a variation of 32,730 p.s.i. with some notch effect to be added. It is not recommended that such chilling be carried out too often, although an occasional or even weekly accidental chilling can be tolerated. Without the transition member or some equivalent device, stress variations (as indicated in Table VI) could soon cause fatigue failure.

It has been stated earlier in this paper that attachment joints at the top and bottom of the cylindrical shell section would be used as a basis for stress analysis. It is also reasonable to use transition members both above and below the cylindrical section. Thus, if a transition member were welded to the bottom cone of a vessel, it would be less subject to hoop stresses due to the smaller radius. The hoop stresses would be directly proportional to the radius from the vessel centerline normal to the liner. Following this pattern, it might be presumed that the radius at the point of attachment might be at the very bottom of the vessel, close to the discharge nozzle. In such a case, however, axial contraction of the liner could cause a severe stress on the weld owing to the small circumferential length of weld resisting the contraction of a large circumferential area of lining. Attachment of the lining to the vessel at about midheight of the top and bottom cones has been done during the past few years in the Kopparfors vessel linings, with success.

Attachment of the linings to nozzles in the cylindrical or other portions of the vessels should also be considered. It has been stated that the lining is free to expand or contract in areas remote from the immediate area of attachment to the vessel. This is not the case where nozzle liners must be attached to the vessel liners. Again, a severe stress in bending can be presumed since a relatively small circumferential length of weld is required to restrain a large area of surrounding liner material from expanding and contracting radially. Again, it is obvious that a ring-type transition member around a nozzle, welded to the shell and nozzle liner at its inside diameter, and to the liner at its outside diameter, would provide stronger and longer welds combined with a flexing section to produce less bending stress.

A further factor should be considered in digester linings. A very suitable lining for both sulfate and sulfite digesters is austenitic stainless steel. These steels are known to be subject to stress-corrosion cracking, if conditions are sufficiently adverse. The adverse conditions are tensile stress and an unsuitable corrosive environment. Experience in Europe has shown that these linings stand up under digester operating conditions as long as chloride concentrations do not accumulate in the liquor.

Stress Corrosion Reduced

If stress-corrosion cracking were possible, it would be most liable to occur during operating conditions nos. 1, 2, 3, and 4, and less liable after no. 5, hot-water rinsing. Table V shows that radial (or hoop) stresses at the fixing joint and the axial stresses are compressive and therefore do not contribute to possible stress corrosion. The free area hoop stresses are tensile stresses. However, they are actually less than shown by about 0.3 of the simultaneous axial compressive stresses, if Poisson's ratio is applied. Maximum tensile stresses in the free area would then be 8480 p.s.i.

CONCLUSIONS

It is known that the Kopparfors bag-type vessel lining has given excellent service over a period of 10 years. Such repairs as have been necessary have invariably been due to weld failures. It is thought that these failures are associated with overstress or fatigue It seems reasonable to expect that minimum maintenance can be attained by decreasing stresses in the vicinity of the attachment welds, and by careful attention to weld details. The complex stress conditions under which a lining must work have been analyzed and the analysis should make it possible to design details which are structurally adequate for the service.

LITERATURE CITED

1. Holgersson, *et al*, U. S. pat. 2,694,632 (Nov. 16, 1954); Can. pat. 488,767 (Dec. 9, 1952).
2. Schorer, H., *Trans. Am. Soc. Civil Eng.* 93 (1933).
3. U.S.A. Ser. No. 22409; Can. Ser. No. 799051; Swedish Ser. No. 6107-60

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Mill Operation of the R–2 Process for Chlorine Dioxide Generation

H. deV. PARTRIDGE and W. HOWARD RAPSON

Full-scale operation of the R-2 Process on a trial basis at three mills in existing generators has confirmed pilot-plant findings and established the following characteristics: high yield of ClO_2, 95% of theoretical based on $NaClO_3$ fed at both high and low production rates; very simple control based on simultaneous, proportional adjustment of R-2 solution ($NaClO_3$ + NaCl), conc. H_2SO_4, air to generator, and water to absorption tower; ClO_2 production rate strictly proportional to chlorate feed rate, resulting in infrequent need for analytical tests; very rapid response to changes in production rate; the process can be operated in any existing ClO_2 generator and pilot-plant data indicate that ClO_2 can be produced at several times the rated capacity of a particular generator; the cost of ClO_2 is lower in many locations than for other processes, particularly where the acid effluent is usable for tall oil splitting.

The R-2 Process for the continuous production of chlorine dioxide from sodium chlorate using sodium chloride (or HCl) as the reducing agent and sulfuric acid for acidity was described in a previous paper (*1*) and in Canadian and U. S. patents (*2*, *3*).

A diagram of the R-2 Process is shown in Fig. 1.

Three streams are fed continuously into a jacketed reaction vessel: R-2 solution, which is an aqueous solution containing sodium chlorate and sodium chloride in the ratio of 3.2 moles chlorate to 3.35 moles sodium chloride: concentrated sulfuric acid (preferably 66° Bé.); air to stir the reaction mixture and to dilute and remove the ClO_2 and chlorine formed.

The concentrated R-2 solution entering the generator immediately reacts in the strongly acid medium to produce ClO_2 and Cl_2 and the reaction products together with the added sulfuric acid feed are rapidly diluted to the concentrations prevailing in the bulk of the generator fluid because of the violent agitation. When a steady state has been reached by operating continuously at constant feed rates, the composition of the generator liquor and the overflowing effluent have the same composition.

Chlorine and chlorine dioxide are produced in the generator according to the following two overall reactions.

(1) $NaClO_3 + NaCl + H_2SO_4 \rightarrow ClO_2 + {}^1/_2Cl_2 + Na_2SO_4 + H_2O$

(2) $NaClO_3 + 5NaCl + 3H_2SO_4 \rightarrow 3Cl_2 + 3Na_2SO_4 + 3H_2O$

An excess of sulfuric acid, sufficient to maintain the generator liquid at 10*N* H_2SO_4, is required in excess of the stoichiometric amount indicated.

The chlorine dioxide, chlorine, and air from the reactor pass through a packed tower where the ClO_2 and about one-quarter the volume of chlorine produced are absorbed in chilled water. The remaining three-quarters of the chlorine produced leaves the ClO_2 absorption tower and can be picked up as sodium- or calcium hypochlorite bleach in an auxiliary tower or venturi scrubber.

Almost all (98–99%) the sodium chlorate decomposes according to reaction (1), giving 1 mole of chlorine dioxide and one-half mole of chlorine per mole of $NaClO_3$. The remaining 1–2% of sodium chlorate decomposes by reaction (2), producing only chlorine. The efficiency of the reaction, *E*, is the percentage of sodium chlorate decomposed which goes according to reaction (1), and *E* can be calculated readily from the gram atom percentage chlorine in chlorine dioxide as follows:

If 100 gram-moles of sodium chlorate decompose by reaction (1), 100 gram-atoms of chlorine are produced as ClO_2 and 100 gram-atoms appear as chlorine. The gram-atom per cent chlorine in ClO_2 is therefore:

$$\frac{100 \times 100}{100 + 100} = 50\%$$

If 99 gram-moles of $NaClO_3$ decompose by reaction (1), 99 moles of chlorine appear as ClO_2 and 99 as chlorine.

The 1 gram-mole which goes by reaction (2), however, produces 6 gram-atoms of chlorine and no ClO_2.

The gram-atom per cent of chlorine in ClO_2 is therefore:

$$\frac{99 \times 100}{99 + 105} = 48.5\%$$

An extension of this process produces the line in Fig. 2.

An analysis of the gases leaving the generator for

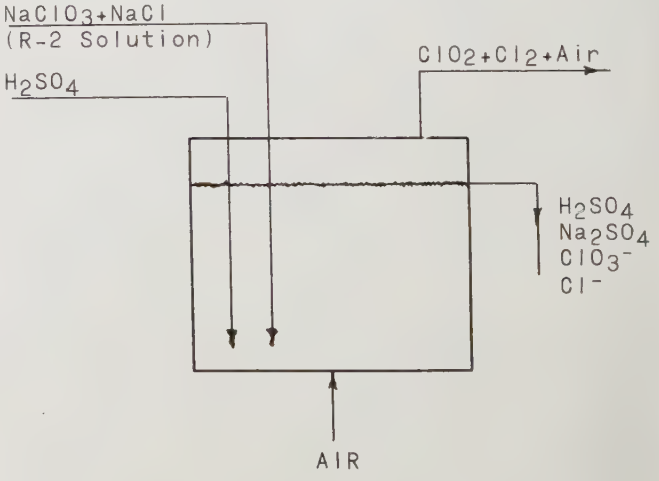

Fig. 1. Diagram of R-2 generator

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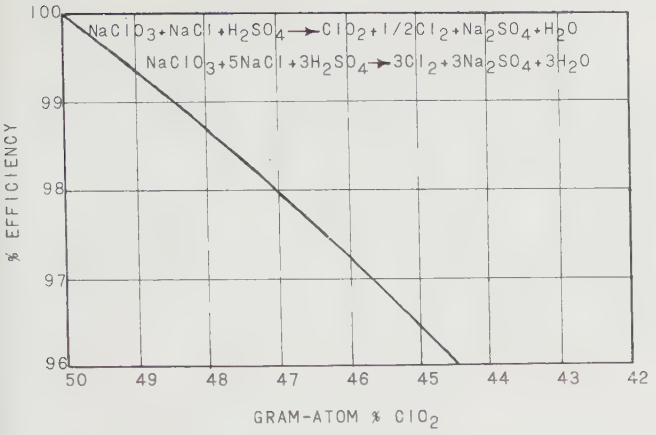

Fig. 2. Efficiency vs. gram-atom per cent ClO_2

ClO_2 and Cl_2 can then be used to calculate the efficiency E.

The per cent decomposed, D, can be calculated from the relationship:

$$\frac{(\text{Moles chlorate in} - \text{Moles chlorate out})}{\text{Moles chlorate in}} \times 100$$

The yield, Y, can then be calculated:

$$Y = D \times E$$

Inventory yield is the only final criterion of mill performance. This is more difficult to determine because of inaccuracy of meters, variations in flows, etc., however, by using calibrated head tanks and carefully standardized meters, it is possible to obtain reproducible yields which check well in most cases with the $(D \times E)$ calculation.

MILL TRIALS OF THE R-2 PROCESS

Three trial runs of the R-2 Process, each of which lasted about a week, were run in 1960. Two of these trials were made in existing SO_2 process generators, the third in an existing methanol process generator.

The purpose of these trials was to demonstrate in actual mill operation the yield and economy of the process found from pilot-plant work and to give mill personnel an opportunity to operate the R-2 Process and assess its potential advantages.

Reactants Used

To facilitate these trials, the R-2 solution was prepared by Hooker and shipped in tank cars to the mill concerned. The sulfuric acid used was the regular mill acid. In two cases this was 66° Bé. acid and in the other 60° Bé. For the R-2 Process, 66° Bé. acid is preferred because it introduces less water into the system and also supplies some heat of dilution to warm the reaction vessel.

Changes in Equipment Required for R-2 Trials

Very few changes in the existing ClO_2 generating equipment were necessary in preparing for the R-2 trials, except to provide for metering R-2 solution and acid at higher rates than for the regular process.

The R-2 reactions produce chlorine which, for the 3 trials, was vented to the atmosphere with no serious problems. In permanent installations the chlorine would be absorbed and utilized.

In order to make rapid changeovers from one process to the other, it is desirable to provide a separate line and flowmeter from the R-2 solution tank to the generator. In changing from the existing processes to the R-2 Process, no difficulties were encountered. It was only necessary to stop the flows of chlorate solution and reducing agent, start the R-2 solution flowing at the required rate and adjust the rate of flow of sulfuric acid to take care of the extra sodium, and to maintain acidity in the generator.

In changing from the SO_2 process to the R-2 Process, there is almost no appreciable change in the generator liquor composition as both processes operate with low-chlorate molarity in the primary generator. In changing from a methanol process, which may operate with 60 to 100 g.p.l. chlorate in the primary generator, to the R-2 Process, a period of hours is necessary to dilute and replace this chlorate before the level drops to the 3 to 6-g.p.l. value of an R-2 generator.

This does not affect the rate of production of chlorine dioxide in any way, but does represent a loss of sodium chlorate during the changeover. If the loss were significant it could be avoided by feeding sodium chloride alone, or methanol, along with the R-2 solution until the chlorate concentration reached the desired level. The conditions required for optimum operation of an R-2 generator over a wide range of production rates have been established by extensive pilot-plant work. These were used as a basis for the mill runs and were found to apply equally well in full-scale operations:

1. The mole ratio of sodium chloride- to sodium-chlorate must be slightly greater than 1.0 to keep the chlorate concentration in the effluent low and to replace the chloride lost by reaction (2). A ratio of 1.05 was used throughout these runs.
2. The acid strength in the generator, particularly in the smaller generators, should be a minimum of $10N$ and preferably $10.5N$ to obtain the maximum decomposition of the chlorate.
3. The temperature of the reaction mixture is not critical. Successful operation was obtained at temperatures ranging from 90 to 150°F.
4. The actual molarities of sodium chlorate and sodium chloride in the R-2 feed solution are important. Solubility data for the system $NaClO_3$–NaCl–H_2O are given in Fig. 3.

The maximum practical combination is $3.30M$ $NaClO_3$, $3.46M$ NaCl. This mixture begins to crystallize at about 40°C. Above these concentrations the temperature at which crystals first form rises very sharply. At $3.35M$ chlorate, $3.52M$ chloride, sodium chloride begins to crystallize out at about 33°C. This actually occurred on the first R-2 trial and low yields of ClO_2 were obtained for the better part of the run, until the reason was found and corrected by addition of several tons of salt and more dilution water to the tank cars. As a result, the R-2 solution concentrations were arbitrarily set at $3.2M$ sodium chlorate and $3.35M$ sodium chloride, and no further trouble has been encountered.

RESULTS

Yields of Chlorine Dioxide by the R-2 Process

The daily inventory yield figures for the mill runs, together with calculated yields from chemical analysis, are shown in Table I.

For test no. 1, only a single day's results are shown. This is because the bulk of the run was made with a

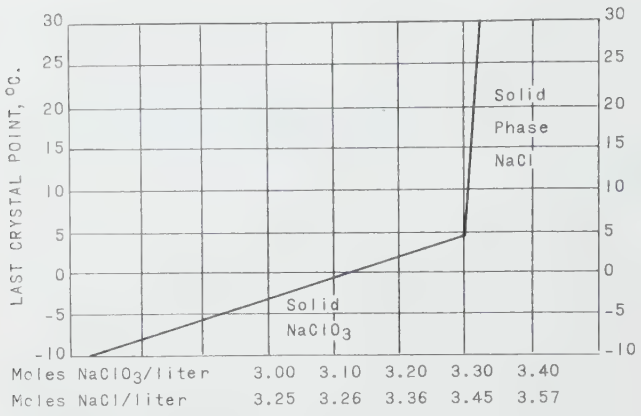

Fig. 3. Equilibrium data for $NaClO_3$-NaCl-H_2O

deficiency of chloride in the R-2 mixture, as previously discussed, and normal operating conditions were obtained for only one full day. In the other two tests four full, consecutive 24-hr. periods were obtained.

The inventory yields shown are essentially the same for both types of generators and are in the range of 95 to 96.0%. Yield figures calculated from the gas analysis and chlorate consumed in the generator, agree very well with the inventory yield figures. At 100% yield, 1.58 lb. of sodium chlorate are required to produce 1.00 lb. of chlorine dioxide. At 95% yield, which is claimed for the R-2 Process, 1.66 lb. of sodium chlorate are required per pound of ClO_2. Existing chlorine dioxide generators are using between 1.75 and 1.90 lb. of sodium chlorate per pound of ClO_2.

The saving in chemical costs with the R-2 Process depend not only on the higher yield of ClO_2 but also on the value of the by-products chlorine, salt cake, and sulfuric acid. Savings are highest for those kraft mills which meet all the following requirements: (1) cook southern pine, (2) bleach about 50% of the production with both hypochlorite and ClO_2, and (3) have a tall oil soap splitting operation.

In such a mill the full valve of the chlorine produced is recovered by converting it to soda or lime bleach. The acid effluent from the generator is fully usable for tall oil soap splitting, and the salt cake from both the generator and the tall oil plant is fully usable as makeup in the kraft mill recovery system.

This is being done now with effluent from existing generators, but the R-2 Process produces more salt cake and more effluent acid which provides large credits per pound of ClO_2 produced. Smaller savings are possible in many other mills where some, but not all, of the above conditions prevail.

Table II shows the comparative chemical costs for the R-2 trials in SO_2-type generators. A yield of 90% is taken for the SO_2 process rather than the actual yields, to avoid disclosing mill figures on the normal operations. The chemical costs shown are not specific for any mill but represent a fair average for the particular area.

Table I. Percentage Yield of ClO_2 by R-2 Process

	SO_2-type generator		*Methanol-type generator*			
	Test no. 1		*Test no. 2*		*Test no. 3*	
Day	*Inventory*	*Analysis*	*Inventory*	*Analysis*	*Inventory*	*Analysis*
1			96.2	95.4	95.2	95.6
2			95.6	94.2	96.2	96.2
3			95.2	95.4	96.2	96.2
4	96.8		95.2	94.3	95.8	95.0
Average			95.5	94.8	95.8	95.6

Table II. Chemical Costs of R-2 Process vs. SO_2 Process
Test no. 2

Per lb. ClO_2	*R-2 Process*			*SO_2 Process*		
	lb.	*¢./lb.*	*¢.*	*lb.*	*¢./lb.*	*¢*
$NaClO_3$	1.65	× 9.0 =	14.85	1.76	× 9.0 =	15.84
NaCl	0.93	× 0.76 =	0.71	...	...	...
H_2SO_4	4.75	× 0.95 =	4.48	1.32	× 0.95 =	1.25
SO_2	...	...	...	0.84	× 4.9 =	4.12
		Total	20.04			21.21
Credit						
Na_2SO_4	2.19	× 1.73 =	3.79	1.3	× 1.73 =	2.25
H_2SO_4	3.6	× 0.95 =	3.40	1.7	× 0.95 =	1.61
Cl_2	0.43	× 3.73 =	1.60	...	...	...
		Total credits	8.79			3.86
		Net chemical cost	11.25			17.35
		Chemical savings—6.10¢./lb.				

In this location the chemical savings amounted to 6.1 cents per pound of ClO_2.

Table III shows the chemical cost comparison for test no. 3 in a methanol-type generator. A yield of 90% was chosen for this comparison rather than the actual yield for the same reason mentioned above. In this location the chemical savings were calculated to be 4.1 cents per pound of ClO_2.

Ease of Operation of the R-2 Process

The overall reactions which generate chlorine dioxide and chlorine by the R-2 Process are the only reactions which can occur, and if the ratio of chlorate-to-chloride in the R-2 solution feed and the normality of acid in the generator are correct, chlorine dioxide in 95% yield will be produced, and once produced it cannot be destroyed by any component present in the system.

The rate of production of ClO_2 is therefore proportional to the rate of feed of R-2 solution to the generator. To start up an R-2 generator it is only necessary to turn on the four feed streams (R-2 solution, acid, air, and water) to the absorption tower simultaneously. Response is almost instantaneous and a new steady state is fully established within 20 min. To double the rate of production of ClO_2, it is only necessary to double the rate of flow of each of the four feeds simultaneously. Again the response is almost instantaneous and a steady state is reached in about 20 min.

To shut down an R-2 generator it is only necessary to stop all flows to the generator simultaneously.

Figure 4 shows the startup of a stripped methanol-type generator on the R-2 Process. The R-2 feeds were turned on simultaneously and set to produce at the rate of 0.9 tons per day. In 10 min. the absorption-tower solution had reached 0.89 tons per day. In

Table III. Chemical Costs of R-2 Process vs. Methanol Process
Test No. 3

Per lb. ClO_2	*R-2 Process*			*Methanol process*		
	lb.	*¢./lb.*	*¢.*	*lb.*	*¢./lb.*	*¢.*
$NaClO_3$	1.66	× 9.0 =	14.94	1.76	× 9.0 =	15.84
H_2SO_4	6.50	× 0.77 =	5.02	3.30	× 0.77 =	2.54
NaCl	0.9	× 1.07 =	0.96	...	× ... =	...
Methanol	...	...	...	0.2	× 4.5 =	0.90
		Total	20.92			19.28
Credits						
Na_2SO_4	2.3	× 1.83	4.20	1.2	× 1.83	2.19
Cl_2	0.4	× 3.91 =	1.57	...	...	...
H_2SO_4	5.1	× 0.77 =	3.84	2.2	× 0.77 ×	1.69
		Total credits	9.61			3.88
		Net chemical cost	11.31			15.40
		Chemical savings 4.1¢./lb.				

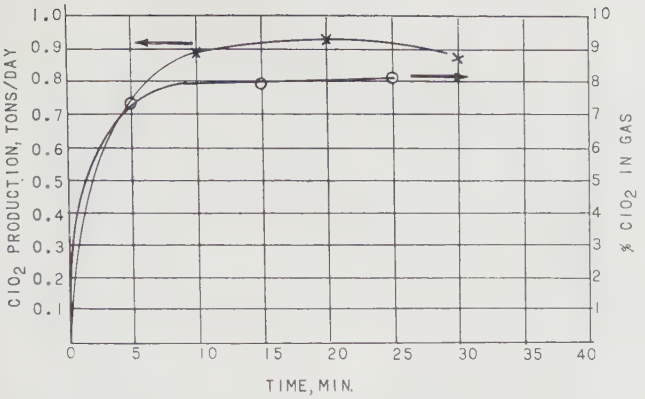

Fig. 4. Startup of stripped methanol-type generator on R-2 process

20 min. it had reached 0.92 tons and was dropping slightly at 30 min. The per cent ClO_2 in the gas followed much the same pattern, leveling off at 8%.

The response to a change in R-2 solution feed rate in an SO_2 process generator is shown in Fig. 5. In this experiment the initial ClO_2 production rate was 1.05 tons/day. The flow of solutions were proportionally increased to give 1.4 tons/day ClO_2. Within 2 min. the production had slightly exceeded the expected rate because of removing some of the ClO_2 stored in the generator, and within 5 min. had practically settled down to the new rate of production.

After running at this rate for about 45 min., the rate of feed of R-2 solution was cut approximately in half. Again the response was very rapid and within 2 min. had dropped below the expected rate and had settled down to a steady state in about 15 min. After running at this rate for about 35 min., the rate of R-2 solution flow was returned to the original level. Response, again, took place within 2 min. and the production of ClO_2 returned to exactly the original rate.

Rate-change studies were carried out during all three runs and confirmed the above findings regarding speed of response and proportionality of ClO_2 production to R-2 solution feed rate.

The process, therefore, lends itself to complete automation and it should be possible to control the plant from a float control, sensing the level of ClO_2 solution in the storage tank.

Testing Required to Operate an R-2 Plant

Because of the simplicity of the reactions, an R-2 plant can be run with very little testing. The ratio of chloride-to-chlorate is fixed when a tank of R-2 solution is prepared. Therefore, testing of the feed solution is necessary only when a tank car of sodium chlorate is unloaded.

The acid normality of the generator liquor changes quite slowly, particularly in the larger reactors, because of the large volume of solution involved. Therefore, only occasional tests should be required for control, perhaps twice per shift.

The chlorate concentration in the effluent naturally reaches a low level if the acidity of the generator liquid is correct and the ratio $NaCl:NaClO_3$ in the R-2 feed solution is greater than 1. Therefore, only occasional tests for effluent chlorate concentration are needed.

The chlorine dioxide concentration in the storage tank will remain constant if the ratio of the flows of R-2 solution and of water to the absorption tower remain fixed. If this ratio can be maintained, very few tests of the ClO_2 solution strength will be required.

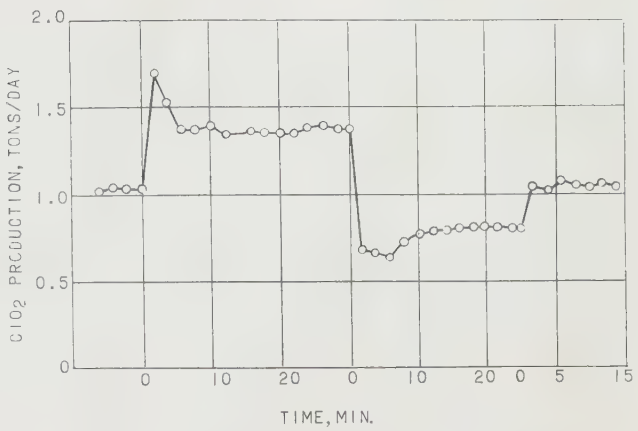

Fig. 5. Change of production rate of R-2 process in SO_2-type generator

When changing production rate, it is not necessary to search for the correct flowmeter settings, so that extra testing during these periods is not required.

It is advisable to test the ClO_2 concentration in the gas stream to the absorption tower at regular intervals, perhaps once per shift, to be certain that the instrument is metering correctly and that the gas concentration is in the safe range of 8 to 12% ClO_2 by volume.

Capacity of an R-2 Generator

Pilot-plant work has shown that the R-2 Process will produce chlorine dioxide in 95% yield, at at least 10 times the rated capacity of an SO_2-type generator and 3 times the rated capacity of a methanol-type generator.

It has not been possible to confirm this completely on a mill scale because of the limitations of the absorption-tower capacity and the meters controlling the flow of reagents and air to the reactor.

However, it has been demonstrated that 98% of the sodium chlorate can be decomposed in the no. 1 reactor of a methanol-type generator with 95% yield, thus effectively doubling its capacity.

Similarly, the output of an SO_2 process generator operating at the limit of capacity was increased 30% by the addition of 15 mole per cent of sodium chloride and sufficient extra sodium chlorate to give the required production (about 15 mole per cent). In this case the additional ClO_2 produced was equivalent to an increase in yield from 80% to 95% plus the ClO_2 produced by the extra sodium chlorate added.

Several mills have been operating through a license agreement under this Hooker patent (*4*) for 6 months to a year, with very satisfactory results. If the pilot-plant data are eventually confirmed in full scale, it will mean that a considerable reduction in the size of ClO_2 generators can be made.

Changes in Equipment Necessary to Convert to R-2 Process

Very few changes are necessary to convert an existing generator to the R-2 Process.

1. The unloading tank must have capacity for 30,000 gal. of R-2 solution to dissolve an 80,000-lb. tank car of chlorate.

2. A source of brine is required. A tank car of sodium chlorate requires 17,500 gal. of saturated brine

and 6,500 gal. of water to make an R-2 solution of 3.2*M* chlorate, 3.35*M* chloride. To prepare the R-2 solution, the brine and water are mixed and heated to 160 to 180°F. in the chlorate unloading tank and circulated through the chlorate car in the usual way. The final solution is then tested for chlorate and chloride content and adjusted, if necessary.

3. It may be necessary to replace the sodium chlorate and acid flowmeters if they are too small to handle the larger volumes of solutions involved.

4. A small, packed tower pump and flowmeter are required to pick up the chlorine produced. For a 6-ton-per-day plant, this tower is 20 in. I.D. with 10 ft. of 1½-in. Raschig rings, irrigated with 25 g.p.m. of 3 to 4% NaOH.

Another alternative method of picking up this chlorine is to pass the vent gases through a venturi, fed with 3 to 4% NaOH or milk of lime from the dilute sodium hydroxide tank on the bleach makeup system. The dilute bleach is recycled to the storage tank. This latter arrangement eliminates the need for an exhaust fan on the ClO_2 towers, and also eliminates a flow controller which would be needed for a tower installation.

CONCLUSIONS

Three mill trials on the R-2 Process in existing generators have demonstrated that this process is a safe, reliable, and highly efficient method for generating chlorine dixoide.

The R-2 Process showed chemical cost savings of up to 6 cents per pound of ClO_2, depending upon mill conditions.

Maximum savings depend on full utilization of the by-products of the reactions, chlorine, salt cake, and sulfuric acid.

The process is simple to operate, responds rapidly to changes in production rate, and lends itself readily to complete automation.

LITERATURE CITED

1. Rapson, W. H., *Tappi* 41, No. 4: 181 (April, 1958).
2. Rapson, W. H., U. S. pat. 2,863,722 (Dec. 9, 1958).
3. Rapson, W. H., Can. pat. 543,589 (July 16, 1957).
4. Rapson, W. H., U. S. pat. 2,936,219 (May 10, 1960).

Received May 3, 1961. Presented at the 46th Annual Meeting of the Technical Association of the Pulp and Paper Industry, held in New York, N. Y., Feb. 20–23. 1961.

Corrosion of Molybdenum-Bearing Austenitic Stainless Steels and Other Alloys in Tall Oil Distillation

HAROLD C. TEMPLETON

Corrosion data from tests run in tall oil distillation plants determine the limits of usability of varying molybdenum content CF-8M—CG-8M alloys and Types 316–317 in such service. Alloy 20, Nionel, Hastelloy C, Inconel, and titanium were also exposed to a number of the test conditions. The corrosiveness of the various test conditions is rated by the weight loss corrosion rate of the 2.4% molybdenum CF-8M alloy, and normal molybdenum content Type 316. Streams having different fatty-acid and rosin-acid contents were used for the tests which were conducted in the liquid and vapor phases. Effect of temperature on corrosion rate is also considered. Streams high in fatty acids are considerably more corrosive than those which are high in rosin acids. In general, the vapor phase is more corrosive than the liquid phase, in both the high fatty-acid and the high rosin-acid streams. Pitting attack occurred on the CF-8M and 316 samples only in vapor-phase tests of high-fatty-acid streams. Streams high in rosin acids can be handled by normal composition CF-8M or Type 316. Liquid-phase streams, high in fatty acids, can usually be handled by normal composition Type 316 or CF-8M alloy, up to 473°F. Vapor-phase streams high in fatty acids require the use of Type 317 or CG-8M alloy. Alloy 20, cast or wrought, has about the same resistance to tall oil distillation service as Type 317 and CG-8M alloy. Hastelloy C and titanium have excellent resistance at all conditions tested.

The subject of corrosion in tall oil distillation has become one of increasing importance because of the large additions to producing capacity during the past several years. The corrosion data presently available indicate the general need for molybdenum-bearing austenitic stainless steels of the 316 type. The need for molybdenum contents higher than are normal in Type 316 material is not clearly defined at present. Tall oil distillation plants have been constructed using both normal and higher than normal amounts of molybdenum in Type 316 steels, (2.4% or 2.5% minimum molybdenum), Type 317 steel, and Hastelloy alloy C.

This work has been done in an attempt to more clearly define the limits where higher-molybdenum-content Type 316 or Type 317 steels become necessary; and to establish the limits where they may be unsuitable. In addition, a number of other corrosion-resistant alloys were exposed to determine the limits of their usability.

This report has been made possible only through the generous cooperation of a number of tall oil distillation plant operators who were willing to expose corrosion test rods in their plants and to release enough information on exposure conditions to make the results meaningful; yet not reveal confidential process information. In some instances the corrosion information came from the files of producers All the producers who collaborated in these tests, or furnished data from their files, have requested that the source of information not be identified.

Table I lists the nominal composition of all the alloys exposed; and the analysis of the samples in Tables VIII, IX, and XIII. Some samples were made from cast materials, in which case the casting material designation is used. Other samples were made from wrought materials, and the wrought material designa-

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Table I. Nominal Analysis

Alloy	C	Cr	Ni	Mo	Mn	Si	Cu	Other
					Per cent			
Type 304	0.06	19.00	9.50	...	1.50	0.50	...	...
CF-8	0.06	19.25	9.25	...	0.75	0.80	...	...
Type 316	0.06	17.00	12.50	2.2	1.50	0.50	...	...
CF-8M	0.06	19.50	10.50	2.4	0.75	0.80	...	...
Type 317	0.06	19.00	13.00	3.2	1.50	0.50	...	...
CG-8M	0.06	19.50	11.00	3.2	0.75	0.80	...	...
Alloy 20	0.05	20.00	28.00	2.5	1.00	0.75	3.50	...
Nionel	0.04	21.50	42.00	3.0	0.75	0.40	2.25	Ti 0.90
Inconel	0.12	13.50	77.50	...	0.80	1.00	...	Fe 6.0
Monel	0.12	...	67.00	...	0.75	1.25	30.00	Fe 1.25
Hastelloy B	0.06	...	Bal.	28.0	0.85	1.00	...	Fe 5.0, V 0.40
Hastelloy C	0.10	16.50	Bal.	17.0	0.75	0.90	...	Fe 5.5, W 4.25
Hastelloy F	0.04	22.00	45.00	6.5	0.75	1.50	...	Cb 2.0
CH 10 (25-12)	0.06	24.00	13.50	...	0.80	0.80	...	...
25-10 Mo (cast)	0.07	25.00	10.00	3.0	0.75	0.85	...	...
Reg. Incoloy	0.04	20.00	32.00	...	0.75	0.35	...	...
Incoloy 901	0.05	13.00	42.50	6.0	0.45	0.40	...	Ti 2.5, Al 0.25
Incoloy 804[a]	...	30.00	42.00	...	...	...	...	Fe-Bal.
2S Aluminum							Commercially pure aluminum	
RC 70-Ti							Commercially pure titanium	
Composition of Samples Exposed to Conditions given in Tables VIII, IX, and XIII								
Type 304	0.07	18.4	9.70	0.3	1.40	0.52	0.22	...
Type 316	0.06	18.0	13.40	2.2	1.50	0.48	0.15	...
Type 317	0.05	18.6	12.60	3.0	1.20	0.50	...	...
U. S. S. Corex	0.07	17.2	24.80	0.9	1.60	0.70	1.30	Ti 0.43

[a] Developmental alloy.

tion is used for these. Where specific analysis of certain specimens exposed was of interest, the analysis is in the corrosion data tables.

Corrosion rates are reported as penetration in mils per year, computed from weight loss during exposure. Additional comments concerning pitting attack, intergranular attack, or other conditions observed, are included in the corrosion data tables.

To correlate the corrosion data, we have classified the tests on the basis of the dominant material in the process stream; i.e., as high in fatty acids or high in rosin acids.

HIGH-FATTY-ACID STREAMS

Table II is a summary of the results from all the streams classified as predominantly fatty acid, listing those materials of primary interest that were exposed on most of the test rods. The test results are arranged in the order of decreasing corrosion rates (weight loss) on the wrought Type 316 sample or the 2.4% molybdenum cast CF-8M sample. The complete test results from each of the exposures are given in Tables III to IX.

The most severe attack occurred in a stream of 65% fatty acids and 35% rosin acids in the liquid phase, at 509°F (Table III). The inhibiting effect of increasing the molybdenum content in reducing corrosive attack is sharply indicated by this test. Corrosion was uniform and no pitting was found on any of these samples. Figure 1 shows the appearance of the 25% chromium–12% nickel sample, and the samples of the three molybdenum levels of the cast, 18% chromium–8% nickel material after exposure. All these samples were of equal diameter and thickness before exposure. The absence of pitting probably results from this being a liquid-phase test; some of the vapor-phase tests, discussed below, produced pitting. An earlier test* in this same stream included a sample of cast alloy 20, which showed a corrosion rate of 27.5 mils per year, and the 2.5% molybdenum CF-8M showed a corrosion rate of 34.2 mils per year. A molybdenum level of 3.0% is necessary to resist corrosion in this service.

The next most severe attack occurred in a stream of 93% fatty acids, 4–5% rosin acids in the vapor phase

* Templeton, H. C., *J. Amer. Oil Chem. Soc.* 36, No. 3: 127–129 (March 1959).

Table II. Summary of Tests in High-Fatty-Acid Streams*

Service conditions	Type 316	CF-8M 2.4% Mo	CF-8M 2.75% Mo	Type 317	CG-8M	Alloy 20	Hastelloy C
	Penetration, mils per yr.						
65% Fatty acids, 35% rosin acids, liquid phase, 509°F., Table III	...	33.1	10.6	2.3	3.8	...	Nil
93% Fatty acids, 4–5% rosin acids, vapor phase, 425–435°F., Table IV	...	7.8 Very uneven attack	3.3 Uneven attack	...	0.7	1.3 Uniform 1 side Pitting 1 side	Nil
90–93% Linoleic-oleic acids, <1% rosin acids, vapor phase, 471–488°F., Table V	...	3.3 Pitting	0.5 Light pitting	Nil	0.1 Few pits	Nil	Nil
97% Fatty acids, 25% vapor- and 75% liquid phase, 455–473°F., Table VI	2.0	...	...	0.1	...	0.6	0.5
55% Fatty acids, 41–43% rosin acids, liquid phase, 400°F., Table VII	...	Nil	...	...	...	Nil	...
Alternately: (liquid phase) fatty acid intermediates, 400°F., fatty acids, 380°F., Table VIII	Nil	...	...	0.1	...	...	...
Alternately: (liquid phase) reflux fatty acid intermediates, 380°F. Reflux fatty acid unsaturates, 360°F., Table IX	0.08	...	...	Nil	...	...	...

[a] Corrosion is uniform in character unless noted.

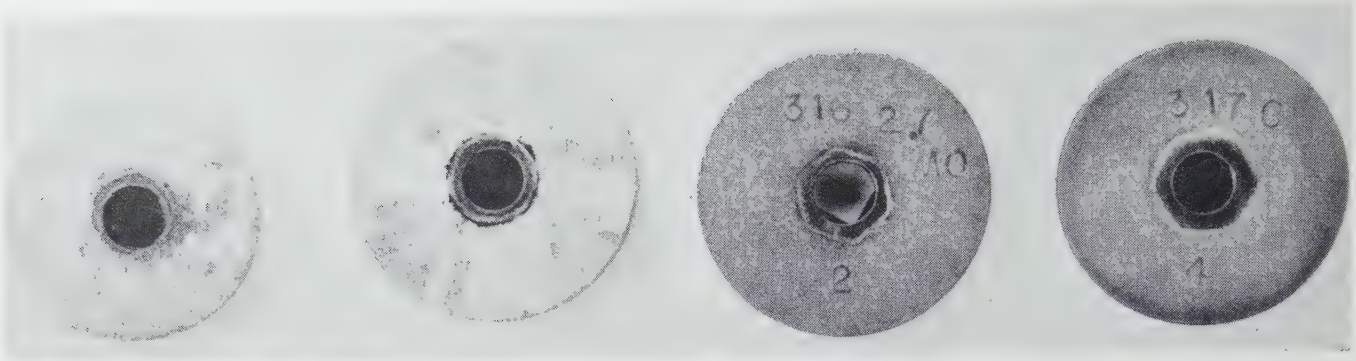

Fig. 1 Tall oil distillation

65% fatty acids–35% rosin acids, 509°F—2400 hr.

Fig. 2 Tall oil distillation

90–93% linoleic-oleic acid (<1% rosin) vapor and liquid+steam, 471-488°F., 1584 hr.

at 425–435°F (Table IV). Increase in molybdenum content reduced corrosion rates on the CF-8M and CG-8M samples by about the same ratio as in the test given in Table III. This test is unusual because of the uneven attack noted on the 2.4% and 2.75% molybdenum CF-8M samples, and to a much lesser degree on the cast alloy 20 and Nionel samples. Attack was uniform and at a low rate on the CG-8M sample. The aluminum sample was severely attacked and its $^3/_8$-in. thickness completely penetrated. Even though the weight-loss corrosion rates on the 2.4% and 2.75% molybdenum CF-8M samples are not excessive (7.8 and

Table III. Tall Oil Fractionating Tower

Solution: 65% fatty acids + 35% rosin acids—liquid phase; temperature: 265°C. (509°F.); exposure: 2400 hr.

Spec no.	*Alloy*	*Penetration, mils/yr.*	*Remarks*
1	CF-8M (2.4% Mo)	33.1	Heavy uniform attack
2	CF-8M (2.7% Mo)[a]	10.6	Moderate to heavy uniform etch
3	Type 317	2.3	Attack varies from light to moderate etch
4	CG-8M[b]	3.8	Moderate uniform etch
6	25-10 Mo (cast)[c]	32.6	Heavy uniform attack
6	25-12 S (cast)	198.6	Very heavy uniform attack—$^1/_6$th original thickness
7	Inconel	16.8	Heavy uniform attack
8	Hastelloy B (cast)	0.0	Very light etch
9	Hastelloy C (cast)	0.0	Bright and clean

	Cr	*Ni*	*Mo*	*Mn*	*Si*	*C*
[a] Composition, %: CF-8M (2.7% Mo)	18.40	13.05	2.69	0.88	1.09	0.043
[b] CG-8M	18.52	13.14	2.97	0.91	0.98	0.042
[c] 25-10 Mo			3.30			

Table IV. Distillation Column

Solution: 93% tall oil fatty acids 4–5% rosin acids; temperature: 425–435°F.; phase: vapor; exposure: 5808 hr.

Alloy	*Pentration, mils/yr.*	*Remarks*
CF-8M (2.4% Mo)	7.8	Spotty attack ranging from light to heavy. Heaviest attack under Hastelloy C spacer and on one edge of sample
CF-8M (2.75% Mo)	3.3	Light to moderate attack, except for edge on one side
Alloy 20 (cast)	1.3	Moderate uniform etch on one side. Other side had many uniformly spaced, very small pits
Nionel	1.7	Many uniformly spaced, very small pits. Heavy attack under Hastelloy C spacer in bolt hole
CG-8M	0.7	Light overall etch, except for few small areas which showed no attack
Monel	6.1	Moderate to heavy even attack
Inconel	11.0	Heavy rough attack
28 Aluminum	1.9	Very uneven attack ranging from light etch to complete penetration of specimen ($^3/_8$" thick)
RC 70-Titanium	0.0	No evidence of attack
Hastelloy C (cast)	0.0	No evidence of attack

3.3 mils per year, respectively); 3.0% molybdenum material should be selected because of the uneven attack on the lower molybdenum alloys.

The next most severe attack occurred in a stream of 90–93% linoleic-oleic acids in the vapor phase at

Table V. Fractionating Tower Reboiler Vapor Nozzle

Solution: tall oil fatty acids—90–93% linoleic-oleic acids (<1% rosin); temperature: 471–488°F. (av. 475°F.); phase: Vapor and liquid + steam—1:0.01:5.0; velocity: 62 ft. per sec.; exposure: 1584 hr.

Alloy	*Penetration, mils/yr.*	*Remarks*
CF-8M (2.4% Mo)	3.3	Heavy overall shallow pitting.[a] Heaviest around and under 316 washer
CF-8M (2.75% Mo)	0.5	Heavy pitting in area of 316 washer. Remainder of specimen pitted, but much less than CF-8M (2.4% Mo) specimen
Alloy 20 (cast)	0.0	Bright and clean. No evidence of attack
Inconel	17.3	Very heavy overall pitting attack
Hastelloy C (cast)	0.0	Bright and clean
Nionel	0.0	Bright and clean
CG-8M	0.1	Slight pitting in area of 316 washer
Type 317	0.0	Bright and clean
RC 70-Titanium	0.0	Bright and clean

[a] All pitted specimens had heavy tenacious coating.

Table VI. Tall Oil Fractionation Reboiler

Test location: 6 in. above outlet of reboiler; solution: 97% tall oil fatty acids, 1.5% rosin acids, 1.7% unsaponifiables; temperature: 455–473°F.; phase: 75% Liquid, 25% vapor. No aeration, high agitation; exposure: 50 days

Alloy	*Penetration, mils/yr.*
Type 316 (2.22% Mo)	2.0
Type 317 (3.34% Mo)	0.1
Inconel	31.0
Alloy 20 (wrought)	0.6
Nionel	0.7
Incoloy 901	0.5
Incoloy 804	34.0
Regular Incoloy	105.0
Hastelloy F	0.4
Hastelloy C	0.5

Table VII. Intermediate Rosin Stream

Solution: 55% tall oil fatty acids, 41–43% rosin acids; temperature: 400°F.; phase: liquid; exposure: 5808 hr.

Alloy	*Penetration, mils/yr.*	*Remarks*
CF-8	0.0	Bright and clean—5 to 6 fairly large pits located at outer edge of Teflon spacer
CF-8M	0.0	Bright and clean. No evidence of attack
Alloy 20 (cast)	0.0	Bright and clean. No evidence of attack
Monel	1.8	Medium to heavy even etch
2S Aluminum	0.0	Bright and clean. No evidence of attack

Table VIII. Distillation Column

Solution and temperature: alternately—fatty acid intermediates at 400°F., tall oil fatty acids at 380°F.; phase: liquid; exposure: six months

Alloy[a]	*Penetration, mils/yr.*	*Remarks*
Type 304	118.0	Severely corroded
Type 316	0.0	...
Type 317	0.1	Very slight attack
U. S. S. Corex	0.0	Minute pits. Intergranular attack to one-grain depth

[a] All specimens in as-welded condition.

Table IX. Distillation Column

Solution and temperature: alternately—reflux fatty acid intermediates at 380°F., reflux fatty acid unsaturates at 360°F.; phase: liquid; exposure: six months

Alloy[a]	*Penetration, mils/yr.*	*Remarks*
Type 304	32.00	Even attack
Type 316	0.08	
Type 317	0.00	...
U. S. S. Corex	0.00	Minute pits. Intergranular attack one-grain depth

[a] All specimens in as-welded condition.

Table X. Fractionation Reboiler

Test location: 6 in. above outlet of reboiler; solution: 91–92% tall oil rosin acids, 1.5% fatty acid, 6% unsaponifiables; temperature: 482–534°F.; phase: 75% liquid, 25% vapor. No aeration, high agitation; exposure: 69 days

Alloy	*Penetration, mils/yr.*
Type 316 (2.22% Mo)	0.2
Type 317 (3.34% Mo)	0.1
Inconel	58.0
Alloy 20 (wrought)	0.2
Nionel	0.2
Incoloy 901	0.2
Incoloy 804	0.3
Regular Incoloy	0.4
Hastelloy F	0.1
Hastelloy C	0.1

Table XI. Distillation Column

Solution: tall oil, 75% to 80% rosin content; temperature: 510–520°F.; phase: vapor; exposure: 4168 hr.

Alloy	*Penetration, mils/yr.*	*Remarks*
CF-8M (2.4% Mo)	2.3	Moderate to heavy even etch
CF-8M (2.75% Mo)	1.2	Light to moderate even etch
CG-8M	0.6	Light even etch
Alloy 20 (cast)	0.4	Light even etch
Nionel	0.6	Light to moderate even etch
Monel	5.0	Heavy uneven attack
Inconel	41.8	Very heavy smooth attack
2S Aluminum	0.1	Very light even etch
RC 70-Titanium	0.1	Very light even etch
Hastelloy C (cast)	0.0	Bright and clean. No evidence of attack

Table XII. Distillation Column

Solution: tall oil, 50 to 55% rosin content; temperature: 440–450°F.; phase: vapor; exposure: 4168 hr.

Alloy	*Penetration, mils/yr.*	*Remarks*
CF-8M (2.4% Mo)	2.0	Moderate to heavy even etch
CF-8M (2.75% Mo)	1.0	Moderate even etch
CG-8M (3.1% Mo)	0.1	Very light even etch
Alloy 20 (cast)	0.4	Light to moderate spotty etch
Nionel	0.3	Light even etch
Monel	4.9	Heavy uneven attack
Inconel	18.6	Very heavy even attack
2S Aluminum	0.1	Very light even etch
RC 70-Titanium	0.0	Very light even etch
Hastelloy C (cast)	0.0	Bright and clean. No evidence of attack

Table XIII. Distillation Column

Solution and temperature: alternately—distilled tall oil at 480–500°F., tall oil rosin acids 480–525°F.; phase: liquid; exposure: 6 months

Alloy[a]	*Penetration, mils/yr.*	*Remarks*
Type 304	118.00	Severely corroded
Type 316	0.00	...
Type 317	0.04	...
U. S. S. Corex	0.36	Many minute pits. Corroded area at top of specimen. Intergranular attack to one-grain depth

[a] All specimens in as-welded condition.

471–488°F (Table V). Again we see the reduction in attack with increase in molybdenum content in the CF-8M and CG-8M alloys. The attack on these samples was primarily from pitting. Figure 2 shows the decrease in pitting attack on the CF-8M and CG-8M alloys after exposure. The pitting is almost wholly eliminated on the CG-8M alloy. The wrought 317 sample was free from pitting. Because of the pitting at the 2.4% and 2.75% molybdenum levels, material of 3.0% minimum molybdenum should be selected for this service.

The test in 97% fatty acids, exposed to 25% vapor and 75% liquid at 455–473°F. (Table VI), showed a corrosion rate on the 316 sample about two thirds of that shown in Table V. No pitting was noted in this test. Increase in molybdenum content from 2.22% to 3.34% reduced the corrosive attack, the same as in the other high-fatty-acid streams where appreciable corrosion occurred. Type 316 of normal composition would be suitable for this service.

The 55% fatty-acid stream in the liquid phase at 400°F. (Table VII), showed negligible corrosive attack on the 2.4% molybdenum CF-8M sample. Even the CF-8 sample showed no overall attack, only a few large pits under the Teflon spacer used in assembling the test rack. The use of CF-8 or 304 alloys should be avoided because of pitting attack. Type 316 of normal composition should be selected.

The streams of Table VIII and IX showed negligible corrosive attack on both the 316 and 317 samples. The 304 alloy sample was severely corroded by these two streams. The samples exposed in these two tests (complete composition shown in Table I) were all in the as-welded condition. No significant attack was found in the area of the welds. Intergranular attack, about one grain in depth, was noted on the Corex samples. Normal composition type 317 would be suitable for these services.

Conclusions

1. The vapor-phase streams are more corrosive than the liquid-phase streams, except for the results from one test (Table III).

2. Pitting attack was found only in vapor-phase tests on CF-8M and 316 alloys.

3. Temperature is obviously a factor in the corrosive attack, but the data do not permit definite conclusions. Severe corrosion on the CF-8M sample at 2.4% molybdenum was found only in the liquid phase at 509°F while the corrosion was minor in the liquid-vapor phase at 455–473°F. Severe corrosion on normal molybdenum CF-8M samples was found in vapor-phase tests at temperatures as low as 425°F. to as high as 488°F. Negligible corrosion was found on normal molybdenum CF-8M and 316 samples in the liquid phase at temperatures below 400°F.

4. Inceasing molybdenum content in the CF-8M/-CG-8M, and 316/317 alloys reduced corrosion rates in every case. Pitting attack, if present, was similarly reduced with increase in molybdenum content in those alloys.

HIGH-ROSIN-ACID STREAMS

The corrosion test results in the streams classed as high in rosin acids are shown in Tables X through XIII. The high-rosin-acid streams are much less corrosive than the streams classified as high in fatty acids.

The most severe corrosive condition, also based on the corrosion rate on either the Type 316 or the 2.4% molybdenum CF-8M sample, was found on the exposure to 75–80% rosin acid at 510–520°F. in the vapor phase (Table XI). The test in 50–55% rosin acids at 440–450°F., also in the vapor phase, Table XII, shows corrosion rates almost as high as those obtained in the test given in Table XI. The lower temperature of the stream in Table XII, with consequent less corrosive tendency, is probably balanced by the increase in the more corrosive fatty acids, when compared to the results of Table XI. CF-8M alloy with 2.4% moybdenum is suitable for use in either of these services.

The 25% vapor-75% liquid test in 91–92% rosin acids at 482–534°F., Table X, gave corrosion rates comparable to the alternate exposure in distilled tall oil at 480–500°F. and to rosin acids at 480–525°F. in the liquid phase Table XIII). Corrosive attack was minor on 316 and 317 alloys in both tests. The samples exposed in the test shown in Table XIII (complete composition given in Table I), were all in the as-welded condition. No significant attack was found in the area of the welds. A slight intergranular attack about one grain in depth was noted on the Corex sample. Type 316 would be suitable for these services.

Conclusions

1. The vapor-phase streams are considerably more corrosive than the liquid phase. Comparison of the test results of Table X with the results of Tables XI and XII shows a corrosion rate ten times greater in the vapor phase than in the 75% liquid-25% vapor phase on the 316 and CF-8M materials. Corrosion rates are moderate even in the vapor-phase tests.

2. No pitting attack was noted on the molybdenum-bearing stainless steels in high-rosin streams, in either vapor- or liquid–phase tests.

3. Regular molybdenum content CF-8M and 316 are suitable for handling tall oil rosin acids within the range of exposure conditions covered by these tests.

4 Temperature is probably a factor in corrosion, however the data are inconclusive.

SUMMARY

1. The high-fatty-acid streams are considerably more corrosive than the high-rosin-acid streams.

2. In general, vapor-phase tests are more corrosive than comparable liquid-phase tests.

3. Pitting on CF-8M and Type 316 alloys was found only in vapor-phase tests in high-fatty-acid streams.

4. Increasing temperature produces higher corrosion rates, but the correlation is poor, based on the tests reported here. There are apparently other factors besides stream composition, temperature, and exposure phase, which greatly influence corrosion rates and may be of greater importance.

5. High-rosin-acid streams can be handled by normal-composition Type 316 or CF-8M.

6. High-fatty-acid streams, liquid phase, can be handled by normal-composition Type 316 or CF-8M, up to 473°F.

7. Fatty acid streams, 90–93%, vapor phase, will probably require Type 317 or CF-8M alloy for temperatures between 425 and 488°F.

8. Alloy 20, cast or wrought, and Nionel, have the same resistance to corrosive attack as Type 317 and CG-8M alloys under most conditions, but not in all cases.

9. Hastelloy alloy C and titanium have satisfactory resistance to all conditions of tall oil distillation tested.

10. Some of the other alloys tested, which contain molybdenum, may be suitable for tall oil distillation service. Additional testing is necessary to establish limits of usability for these materials. The molybdenum-free alloys have only limited usage in tall oil distillation service.

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Automatic Heating and Temperature Control of the ASTM D 127 Melting Point Bath

B. R. BLUESTEIN, J. C. EATON, JR., and K. E. GRIFFITHS

Equipment and technique is described for the automatic heating and temperature control of the water bath used in the ASTM D 127-60 (TAPPI Standard T 634 m-56) method for the melting point of microcrystalline wax.

THE ASTM D 127-60 (TAPPI Standard T 634 m-56) method for Melting Point of Petrolatum and Microcrystalline Wax (*1*) is a convenient procedure for controlling the production and quality of microcrystalline waxes. These waxes are used for paper laminations and in wax coatings.

The ASTM method for melting point requires a heating bath which is heated at a definite, prescribed rate. The bath temperature is raised at the rate of 3°F. per min. from 60 to 100°F.; then the temperature is increased at a rate of 2°F. per min., from 100°F. to the melting point of the wax.

When a laboratory technician uses this method he keeps adjusting the heating rate of the water bath manually, whether heated with a gas-burner flame or with an electric heater. Also, the technician must continually check the heating rate with a stopwatch. This double operation requires the close and complete attention of the technician. Therefore, the development of an automatically controlled heating bath relieves the technician of this time-consuming heating operation.

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AUTOMATIC HEATING AND TEMPERATURE CONTROL EQUIPMENT

Several different melting point procedures have been improved by the incorporation of automatic operations. Smit and Kateman (*2*), and Berhenke (*3*), installed automatic recorders for heating curve melting points and for capillary melting points, respectively.

We constructed a 3-liter heating bath for the ASTM D 127–60 method, with the temperature controled and indicated by a cam program controller. This bath is heated by a low-lag cartridge, immersion electric heater controlled by a gas-filled temperature-sensing bulb, which signals the temperature of the water bath to the program controller. The cam in the program controller is cut for the specific heating rate required for the ASTM D 127–60 procedure. Any standard type cam program controller is suitable for this purpose (*4*). Figure 1 is a diagram of the automatic heating and temperature control equipment.

The water-bath temperature is kept uniform by an electric stirrer. The bath contains six standard test tubes, each fitted with an ASTM melting point thermometer having a range of 90 to 260°F., 32 to 127°C. (ASTM Standard E 1, thermometer 61F. or 61°C). This bath may be used for the melting point determination of as many as six different waxes at one time or two different wax samples, each in triplicate. Figure 2 shows the heating bath and program controller.

Table I. Physical Properties of Petroleum Waxes

	Melting point, ASTM D 127–60[a], °F.	*Needle penetration at 77°F., °F. ASTM D 1321–57 T*[b]	*Lovibond color, 2-in. cell*	*Color, ASTM D 1500–58T*	*Saybolt viscosity at 210°F., ASTM D 88–56, S.U. sec.*
Microcrystalline wax					
Wax A	151	41	35Y 1 1/4R	L 1.5	80
Wax B	168	27	35Y 1 1/2R	L 1.5	87
Wax C	180	29	1/2Y	L 0.5	78
Wax D	180	29	20Y 1 1/4R	L 1.5	80
Wax E	187	19	35Y 1 3/4R	L 1.5	83
Wax F	201	5	1 3/4Y	L 0.5	88
Semimicrocrystalline wax					
Wax G	132	19	9Y 1/2R	0.5	47
Paraffin wax					
Wax H	126	41	<1/4Y	L 0.5	38
Wax I	130	17	<1/4Y	L 0.5	39
Wax J	140	11	<1/4Y	L 0.5	40

[a] TAPPI TENTATIVE METHOD T 634 m-56.
[b] TAPPI SUGGESTED METHOD T 639 sm-57.

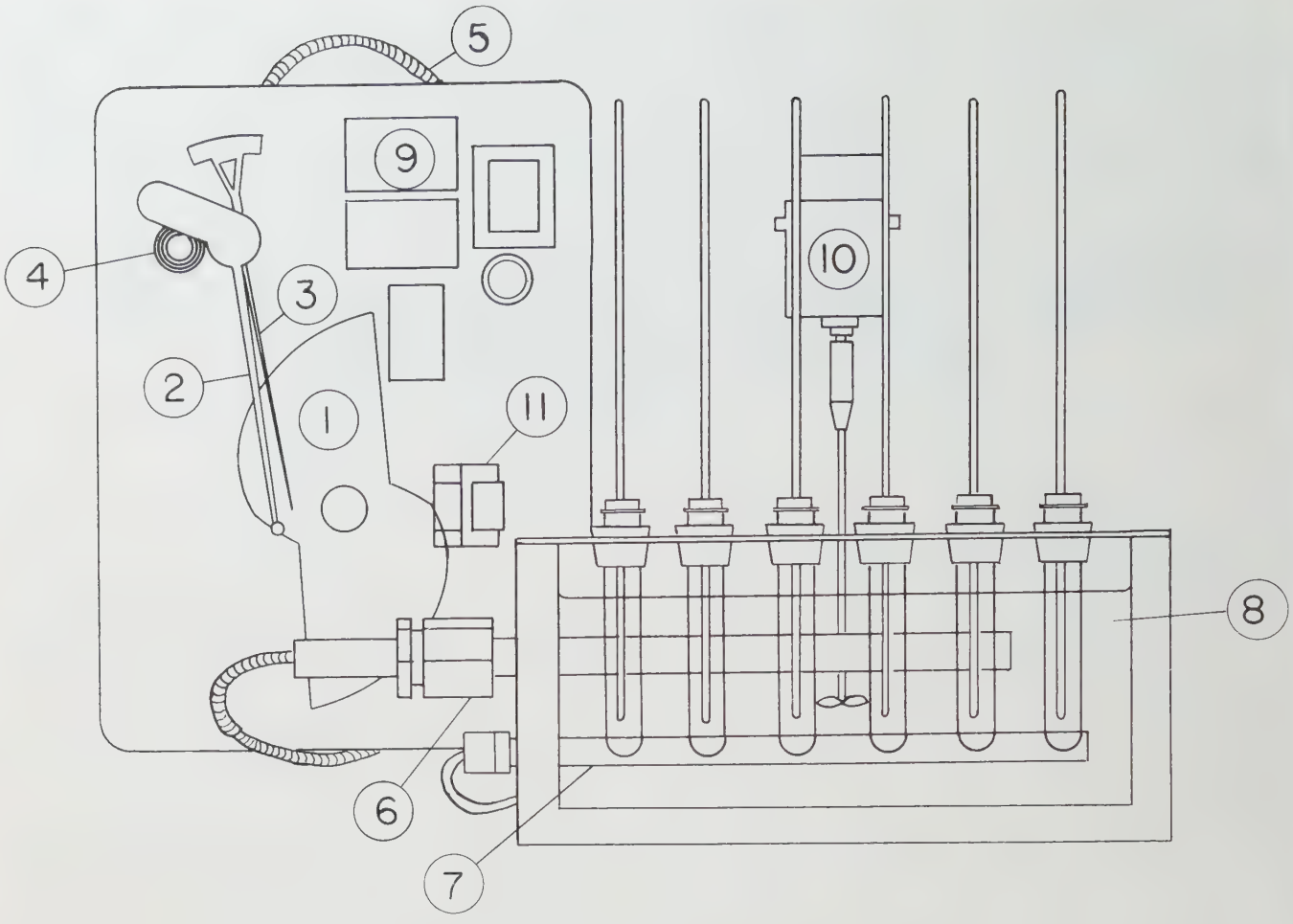

Fig. 1. Automatic heating and temperature control equipment

1. Cam. *2.* Cam follower. *3.* Temperature indicator. *4.* Bourdon tube pressure spring. *5.* Capillary tubing. *6.* Temperature-sensing bulb. *7.* Electric immersion heater. *8.* Water bath. *9.* Relays. *10.* Electric stirrer. *11.* Cam drive motor.

MELTING POINTS

The melting points of various petroleum waxes were determined by the ASTM D 127–60 method, using both the manually controlled heating bath described in this ASTM method and the automatically controlled heating bath described in this article. The properties of the waxes are listed in Table I. Three paraffin waxes and a semimicrocrystalline wax are included in this study, along with six microcrystalline waxes. The melting point values of the waxes determined in the two different types of heating baths are compared in Table II.

Table II. Melting Points of Petroleum Waxes, (ASTM D 127–60)

Sample designation	*Manually controlled heating bath (ASTM D 127)* — *Three determinations, °F.*	*Melting point av., °F.*	*Automatically controlled heating bath (this article)* — *Three determinations, °F.*	*Melting point av., °F.*	*Deviation in m.p. between baths, °F.*
Microcrystalline wax					
Wax A	150.2, 151.0, 152.0	151.1	150.1, 151.5, 150.9	150.8	−0.3
Wax B	167.5, 167.8, 167.5	167.6	168.4, 168.5, 167.8	168.2	+0.6
Wax C	179.0, 180.0, 178.1	179.0	180.0, 180.7, 179.8	180.2	+1.2
Wax D	181.1, 181.9, 180.6	181.2	180.1, 179.3, 179.7	179.7	−0.5
	178.9, 180.2, 179.6	179.6			
Wax E	187.3, 188.0, 187.0	187.4	186.8, 187.2, 187.2	187.1	−0.3
			186.7, 187.0, 187.0	186.9	
Wax F	201.3, 201.4, 201.6	201.4	201.3, 201.2, 202.0	201.5	+0.1
	201.3, 201.4, 201.2	201.3			
Semicrystalline wax					
Wax G	133.0, 131.8, 132.0	132.3	133.0, 132.2, 132.5	132.6	+0.3
	131.8, 132.2, 132.0	132.0	131.9, 131.7, 131.7	131.8	−0.2
Paraffin wax					
Wax H	125.7, 125.7, 125.6	125.7	126.3, 127.7, 126.5	126.8	+1.1
Wax I	130.8, 130.1, 131.2	130.7	130.2, 129.8, 130.0	130.0	−0.7
	130.0. 130.1, 130.6	130.2			
Wax J	140.1, 140.2, 140.2	140.2	140.6, 140.6, 141.1	140.8	+0.6
				Av. devn. =	0.5
				Std. devn. =	0.7

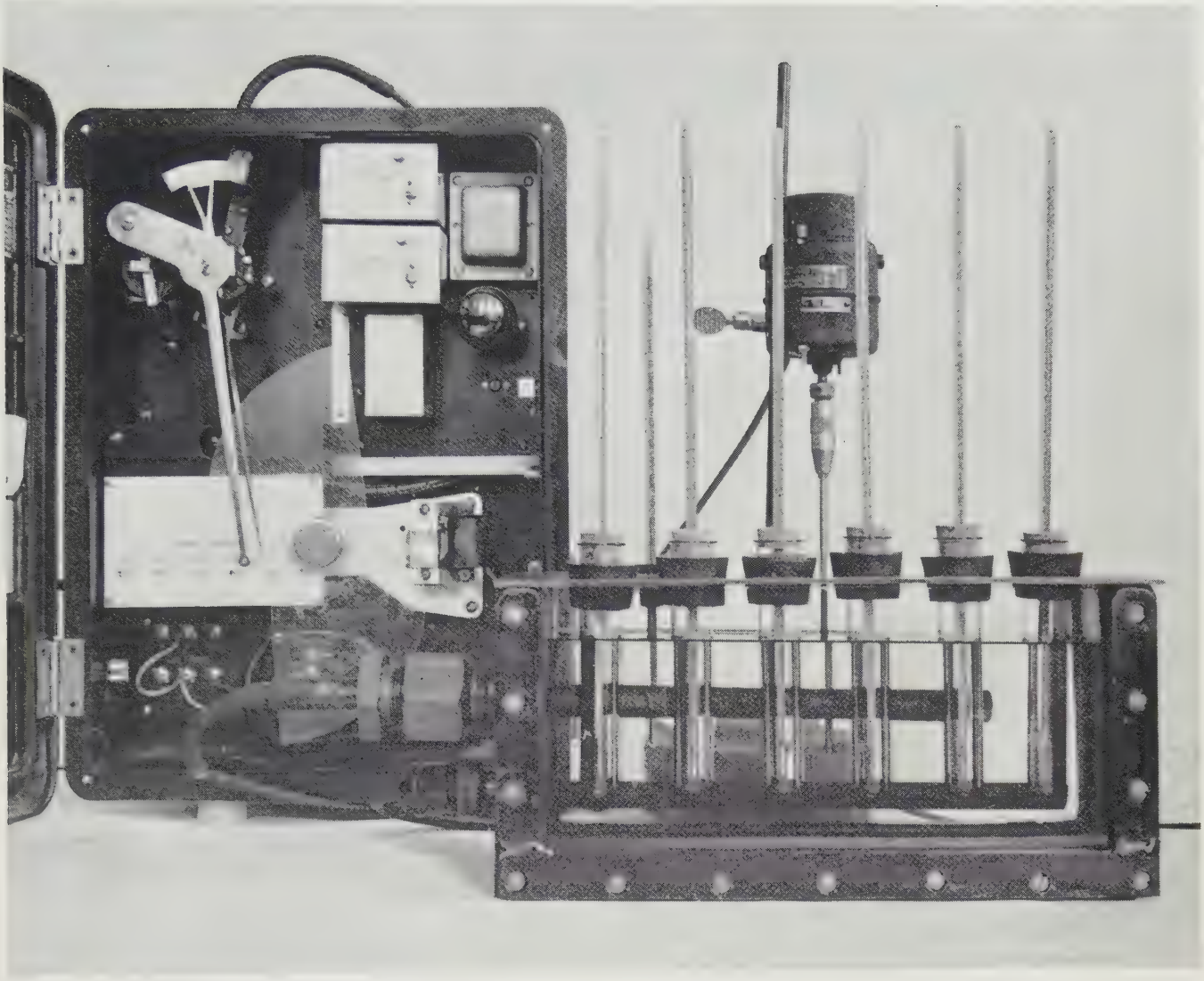

Fig. 2. Heating bath and program controller.

The ASTM D 127–60 method requires that the variation of three individual determinations for one wax should not exceed 2°F., otherwise two additional determinations must be made. The average of three (or five) such determinations is the ASTM D 127 melting point. All of the sets of three determinations for the waxes in Table II had variations less than 2°F. Therefore, using the automatically controlled heating bath did not introduce any additional variation into the ASTM D 127 melting point method.

The melting points (the averages of each three determinations) of each wax determined in the two different types of heated water baths are compared in the last column in Table II as the "Deviation in M.P. between baths, °F." Five of the deviations are negative, showing that the melting points determined in the manually controlled heating bath were higher than those determined in the automatically controlled bath. Six of the deviations are positive, showing the opposite relationship. This balanced distribution of the deviations is evidence that the automatically controlled heating bath does not introduce any systematic error into the ASTM D 127 melting point method.

The ASTM D 127 melting point of each wax, determined in the automatically controlled heating bath, differs from its value determined in the manually controlled bath by an average deviation of 0.5°F. and a standard deviation of 0.7°F. This standard deviation of 0.7°F. between melting points with the different heating baths is much less han the variation of 1.5°F. allowed for the repeatability of the method. Therefore, the use of the automatically controlled heating bath does not introduce any new variable into the ASTM D 127 procedure.

CONCLUSION

The water bath used in the ASTM D 127–60 (TAPPI Standard T 634 m-56) method for the Melting Point of Petrolatum and Microcrystalline Wax can be heated automatically by a cam program controller.

The melting point values of petroleum waxes obtained by using the automatically heated and temperature controlled water bath are the same as the values obtained using the manually controlled heating bath.

LITERATURE CITED

1. Am. Soc. Testing Materials, "ASTM Standards on Petroleum Products and Lubricants," 55 (Oct., 1960).
2. Smit, W. M. and Kateman, G., *Anal. Chim. Acta* 17, 161 (1957).
3. Berhenke, L. F., *Anal. Chem.* 33, 65 (1961).
4. Cameron, R. G., *Instrumentation* 12, No. 3: 8 (1959).

RECEIVED March 23, 1961

New Venturi Scrubber Developments

The Steam-Atomized Venturi

P. H. WEST, H. P. MARKANT, and J. H. COULTER

The ever-increasing demands that the alkaline pulping industry lower particulate matter in stack gas led to a series of pilot plant studies on ways to improve the collection efficiency of the venturi evaporator scrubber. This device had already proved its effectiveness in recovering fumes from the exhaust gases of a chemical recovery unit and in improving the recovery of heat by reducing exit-gas temperature, and has received commercial acceptance since it was first installed at Thilmany Pulp and Paper Co. in May, 1953. The pilot-plant studies have resulted in the development of a steam-atomized venturi scrubber. This paper describes the circumstances leading to its development and presents performance data and operating experience on two commercial installations. The steam atomized venturi scrubber has demonstrated improved collection efficiency at no additional power consumption.

In July, 1947, after considerable pilot-plant investigation, Thilmany Pulp and Paper Co. installed the first full-scale venturi scrubber behind their 120-ton recovery unit. Water was used as the scrubbing medium to collect the salt cake fume from the recovery unit flue gases. The concentration of the water or recirculated salt cake brine solution was maintained at about 30% solids by bleed-off to the direct contact evaporator or mix tank to supplement the salt cake makeup. Chemical recovery efficiency was 85 to 90% with 15–17 in. H_2O pressure drop across the venturi throat and cyclone separator. Thilmany's experience with the full-scale brine scrubbing venturi is described by Collins, Seaborne, and Anthony (*1*) and by Collins and West (*2*).

In the brine scrubbing venturi, the greatest disadvantage lies in the relatively large amount of water that must be evaporated from the brine bleed-off to the black liquor. Therefore, it was a logical step to consider substituting black liquor as the scrubbing medium in place of the water or brine. With the arrangement shown in Fig. 1, two functions are performed in a single piece of equipment: the concentration of the makeup black liquor from the multiple effect evaporators is raised to the desired level for firing by recovery of considerable heat, and a large percentage of the chemicals are recovered from the flue gases.

Based on the above, Thilmany Pulp and Paper Co. specified that a black liquor venturi scrubber be installed behind the 250-ton recovery unit, that went into service in April, 1953. Figure 2 shows the venturi header and throat arrangement. A previous paper (*3*) described the chemical and heat recovery of this equipment in detail. Here again, chemical recovery was 85 to 90%, but with 30-in. H_2O pressure drop as compared to 15 to 17 in. with brine scrubbing. The more viscous black liquor required more power to obtain the same scrubbing efficiency.

The ever-increasing demands that the alkaline pulping industry lower particulate matter in stack gas led to a series of pilot-plant studies on ways to improve still further the collection efficiency of the venturi evaporator scrubber. From these studies came the steam-atomized venturi scrubber. The development work leading to this arrangement, and the comparisons between commercial and pilot-plant tests in each step of the development are presented in this paper.

PILOT-PLANT STUDIES

Objectives

In January, 1957, pilot venturi scrubber investigations were undertaken at Kaukauna, Wis., as a joint effort of the Thilmany Pulp and Paper Co. and The Babcock & Wilcox Co. The purpose was to:

1. Determine the possibility of increasing the salt cake collection efficiency to meet the increasingly stringent air pollution regulations.
2. Determine the relationship of controllable parameters, so that a better insight into venturi operation could be obtained. Such information would be valuable for further improving the performance of existing units.
3. Determine the design point at which each parameter should be set for optimum performance, so that standards for better scrubber design could be set.
4. Develop a venturi scrubber to accomplish maximum salt cake recovery with minimum power costs on practical commercial basis. A unit having 90% collection efficiency with 20-in. H_2O pressure drop, or 95% for 30-in. H_2O pressure drop were the ultimate objectives.

Description of Pilot Unit

Considerable care was exercised in the design of the pilot venturi scrubber. A $^1/_{10}$-scale model of a 250-ton commercial unit consisting of a venturi and cyclone separator was used for all tests. A diagram of this unit is shown of Fig. 3. This test unit was installed in parallel with the existing unit at Thilmany. Flue gas was taken from the parent unit immediately in front of the venturi throat and returned to the flue system at the cyclone outlet. Both high-density (approx. 60% solids) and low-density liquor (approx. 45% solids) were available from the parent unit.

A separate induced-draft fan was used on the pilot unit to extend the range of gas flows and velocities for these tests. Gas flow was controlled by a butterfly damper in front of the induced-draft fan, and gas-weight flow was measured by a venturi flow nozzle located in the hot-gas inlet duct. Gas was sampled before the flow nozzle (station *A*) and after the cyclone separator station *B*). Egg-crate type flow straighteners were used

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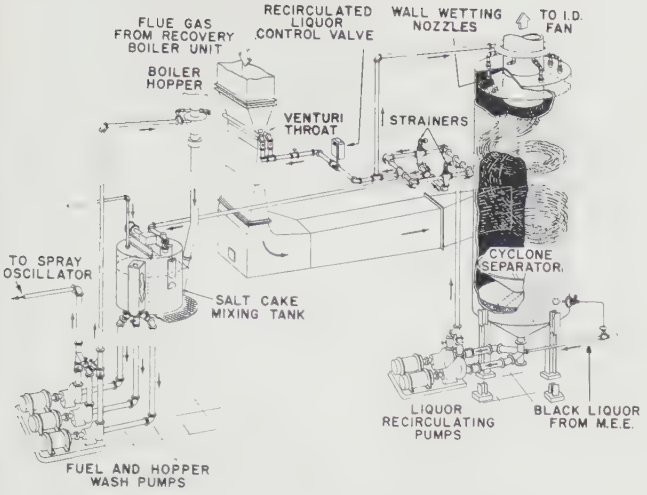

Fig. 1. Venturi evaporator scrubber

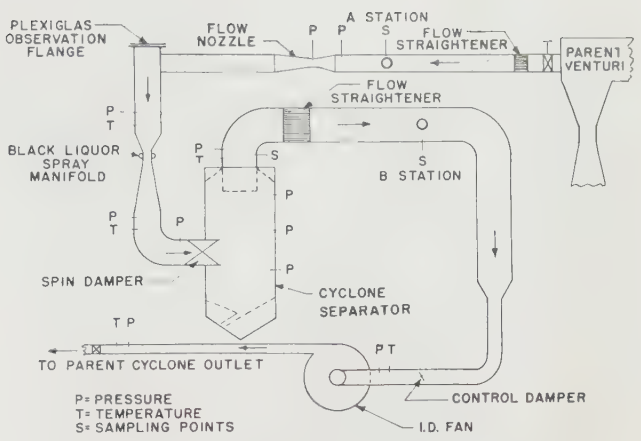

Fig. 3. Diagram of pilot unit venturi scrubber

in front of both sampling points. The gas velocity in the throat was controlled by varying the gas flow through the unit. Further flexibility in the throat velocity was obtained by varying the rectangular throat area from 6 to 20 sq. in.

Black liquor booster pumps were used to increase the range of liquor flows of both high- and low-density liquor to the unit with solids measured with a Bausch and Lomb hand refractometer. A calibration curve, based on oven-dried solids, was developed for this instrument, and it was found to be accurate to within $\pm 1\%$ solids. The venturi spray manifolds were mounted on the long axis of the throat and the flow through them was determined by relating pressure-gage readings to spray manifold calibration curves.

The instrumentation shown on Fig. 3 was of the usual type, with manometers and thermocouples comprising most of the equipment.

The method (*5*) used for determining both the venturi inlet and outlet dust loadings was the same as that commonly used by the Babcock and Wilcox Co. for commercial tests. The apparatus used is shown on Fig. 4, and consists of a special sampling probe designed to permit isokinetic sampling, and a condensing train.

The sodium salt carried by the flue gases from a black liquor recovery unit consist of both fume and aggregate particles. The fume is usually well distributed throughout the gas stream because it is made up of small particles, but the aggregate particles are often affected by the force of inertia and gravity, which complicates the problem of obtaining a representative sample. Therefore, the sampling equipment was designed to minimize this problem by removing the sample at a velocity equivalent to the gas velocity in the flue.

Once a representative sample of the dust-laden gas was obtained, it was analyzed by standard chemical analytical methods. The total sodium in the sample was determined by a flame photometer, and sodium content of the flue gas was calculated from the volume

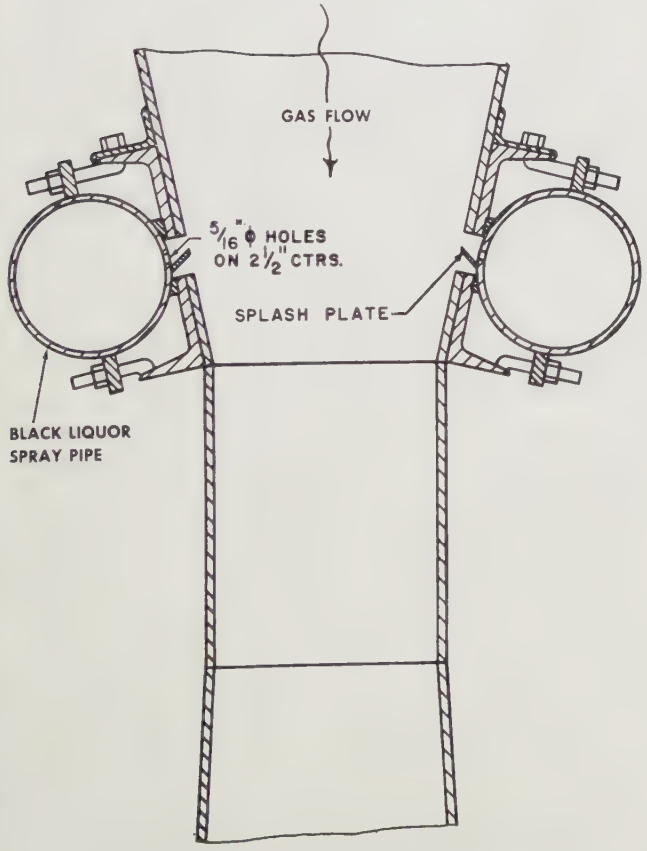

Fig. 2. Venturi evaporator scrubber throat arrangement

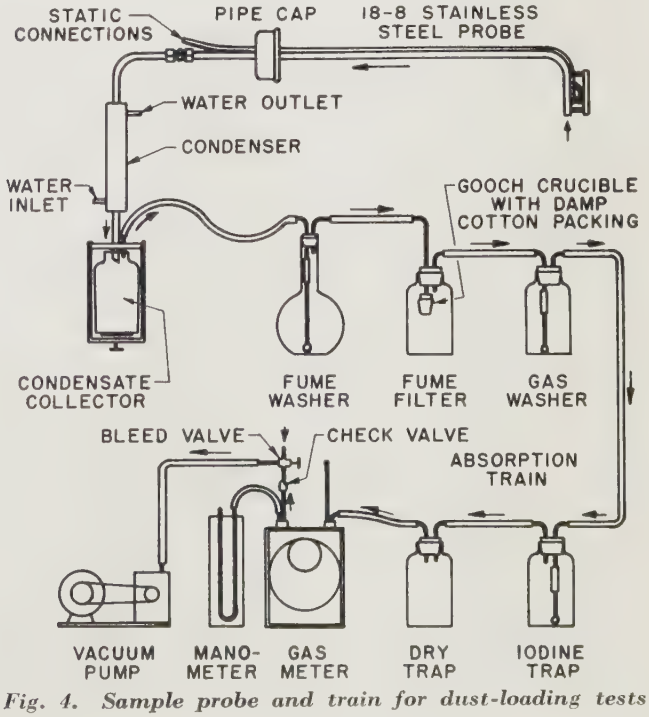

Fig. 4. Sample probe and train for dust-loading tests

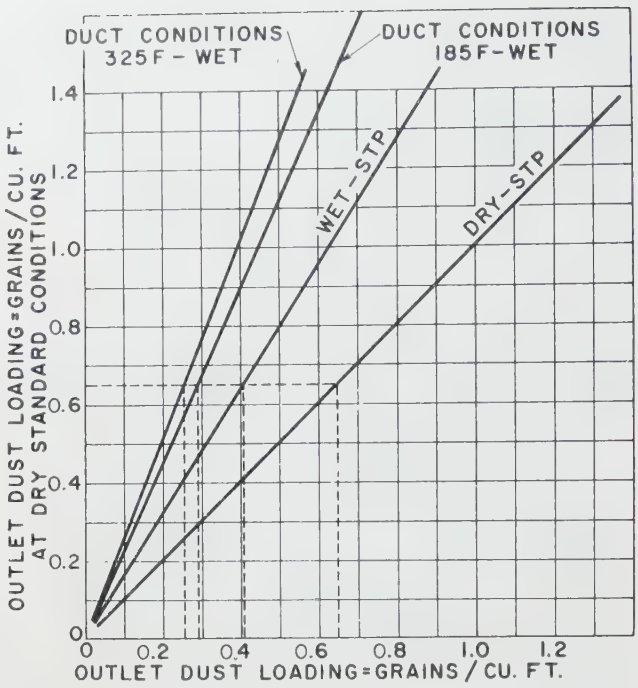

Fig. 5. The effect of base conditions on dust loading

temperature, pressure, and humidity of the gas sample and expressed as sodium sulfate per cubic foot of dry gas at standard conditions.

Since the dust loading on recovery units is often reported at conditions other than the dry standard condition, Fig. 5 shows the significant differences when the dry standard condition is compared to other bases. For instance, a reported outlet dust loading of 0.65 grains per cubic foot at dry standard conditions would be equivalent to the following:

Wet standard conditions	0.41 gr. cu. ft.
Duct conditions @ 185°F	0.29 gr./cu. ft.
Duct conditions @ 325°F	0.26 gr./cu. ft.

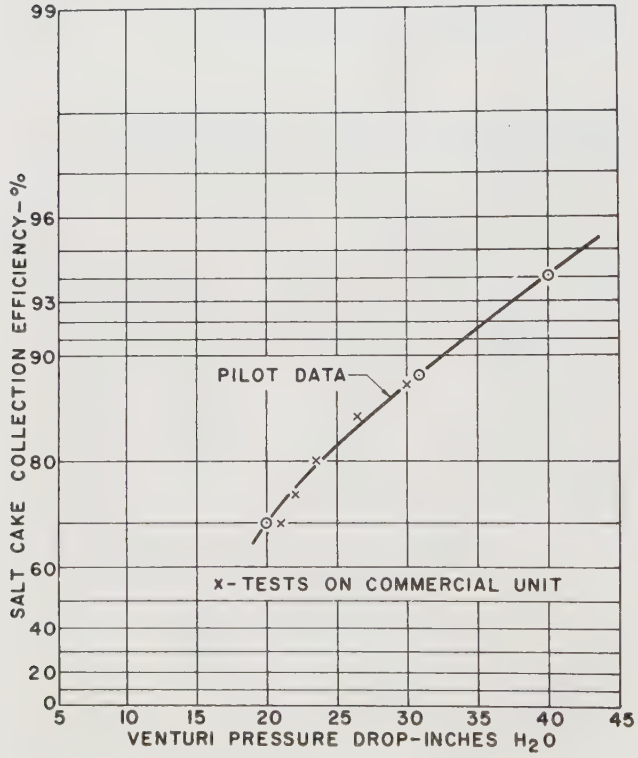

Fig. 6. Comparison of commercial and pilot data single header venturi

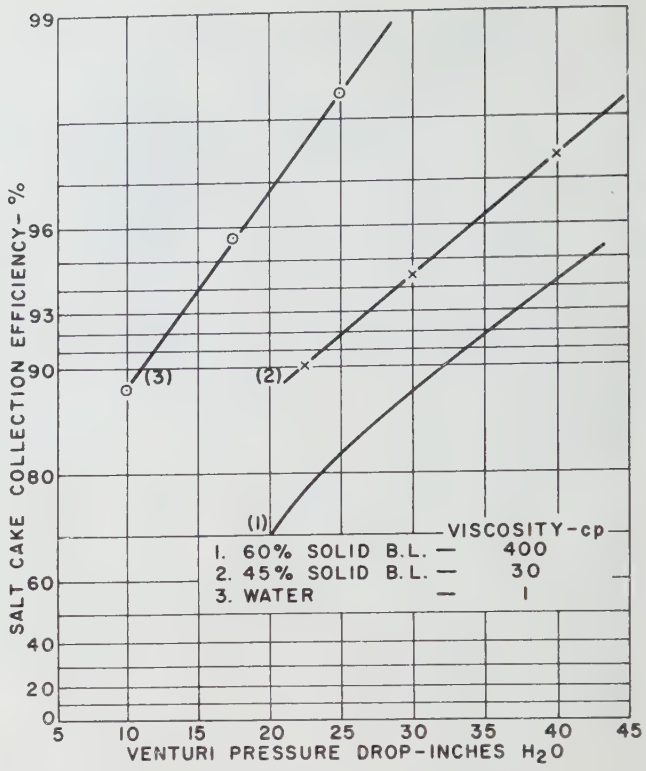

Fig. 7. The effect of fluid properties and venturi pressure drop on scrubbing efficiency in a single heater venturi

It is therefore extemely important, when comparing one piece of equipment with another, that the loading conditions be specified.

TEST PROGRAM

Before any development work could be undertaken with the pilot unit, the relationship between pilot and commercial venturi operation had to be established. This was done by running the pilot unit at conditions similar to the Thilmany venturi; i.e., the gas velocity, liquor-to-gas ratio, and total pressure drop were the same. The pilot venturi pressure drop was then varied from 17.5 in. to 45 in. of water in incremental steps. Venturi inlet and outlet dust loadings were obtained for each change, and the results were plotted as shown on Fig. 6. When it was seen that these results agreed with field data (*3*), the first step was to investigate the effect of fluid properties on collection efficiency.

The Effect of Fluid Properties

Since it was known that venturi performance is largely affected by the particle size of the scrubbing solution, it seemed reasonable to assume that properties such as surface tension and viscosity, would have considerable effect on the performance. To minimize the number of variables, it was decided to test various typical solutions common to the kraft industry rather than fluids that might possess a wider range of properties. Three fluids were chosen for this purpose:

	Viscosity, cp.
Black liquor, 60% solids	400
Black liquor, 45% solids	30
Water (cold)	1

The pilot unit shown in Fig. 3 was operated over a range of pressure drops with each of these fluids. The

inlet and outlet dust-loading data were used to prepare the curve showing the effect of fluid properties and venturi pressure drop on the collection efficiency as shown on Fig. 7. These tests indicated that as the viscosity of the liquid decreased a significant increase in collection efficiency was obtained for the same pressure drop across the venturi throat. Or, as the viscosity of the liquid decreased, the same collection efficiency was

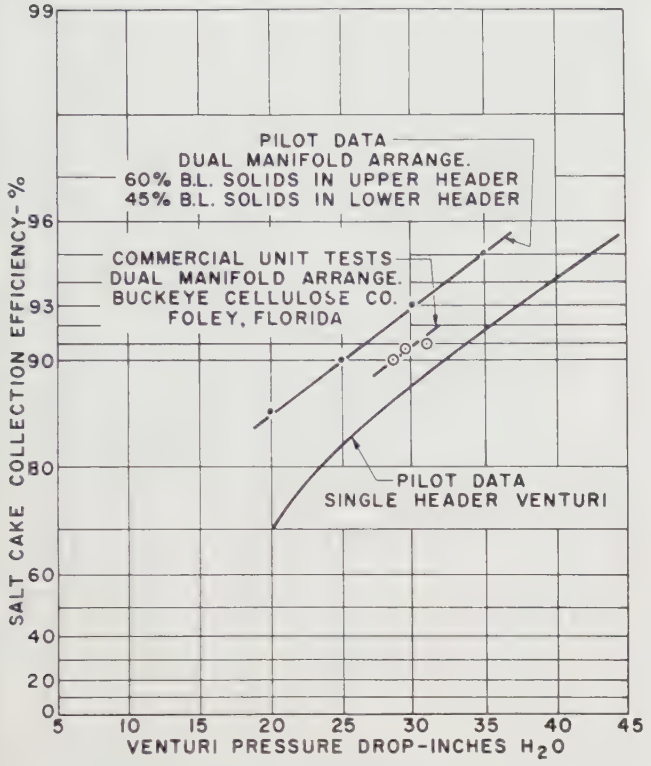

Fig. 8. Comparison of commercial and pilot data, dual manifold arrangement

obtained at a lower venturi differential for less total energy. For example, approximately 32 in. pressure drop was required for a collection efficiency of 90% when using black liquor of 60% solids whereas only 22 in. was required when using black liquor of 45% solids. A still further decrease in pressure drop was realized when using water.

One can therefore conclude that the less viscous the scrubbing medium, the greater the collection efficiency for a given amount of mechanical energy expended; or, less mechanical energy would be required to obtain a given collection efficiency.

This knowledge of the effect of fluid properties on the collection efficiency prompted an investigation on the pilot unit of ways to make maximum use of the 45% liquor from the multiple-effect evaporator. This led to the development of the dual manifold arrangement.

The Dual Manifold Venturi

Pilot Studies. All black liquor venturi scrubber installations prior to this experimental work were designed with a single set of spray manifolds installed at the venturi throat (see Fig. 2). The recirculated liquor from the cyclone separator sump was sprayed through these headers, and the makeup liquor from the evaporators was added to the sump. To determine the benefits that could be obtained by spraying this 45% liquor into the venturi throat to take advantage of its low viscosity, the pilot unit was modified to a dual manifold arrangement. With this arrangement, the low viscosity liquor normally sent to the cyclone sump was introduced into a separate set of headers at the throat, downstream from the recirculated liquor headers.

The effectiveness of this arrangement is shown in Fig. 8. When compared with the single header venturi, it

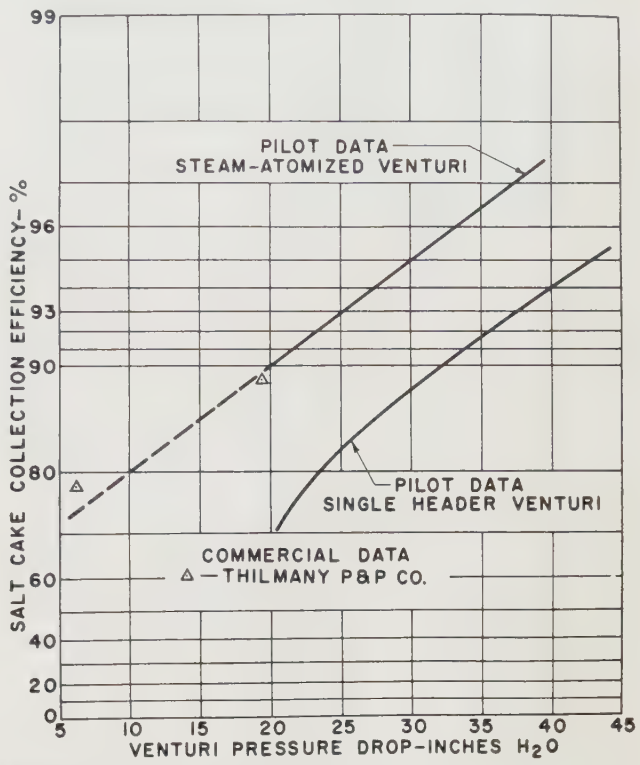

Fig. 9. Comparison of commercial and pilot data, steam-atomized venturi

will be seen that there is a significant increase in collection efficiency for the same pressure drop, or that for a given efficiency the pressure drop was materially reduced.

Commercial Evaluation of the Dual Manifold Venturi. The first commercial dual manifold venturi was installed late in 1958 on the 550-ton recovery unit at Buckeye Cellulose Corp., Foley, Fla. (*4*). The results obtained are compared with pilot data in Table I and Fig. 8. As shown, a scrubbing efficiency of 91% was obtained with a pressure drop of 30 in. of water across the venturi throat.

Since all tests on both the pilot unit and full-scale installations had indicated that the fluid properties had a significant effect on collection efficiency and energy requirements, it was felt that if the particle size of the fluid injected into the venturi throat could be reduced, the overall performance of the venturi could be still further improved. Various methods for atomizing the liquor were evaluated on the pilot unit, which led to the development of the steam-atomized venturi.

The Steam-Atomized Venturi

Pilot Studies. The dual manifold pilot plant arrangement was modified so that the recirculated black liquor would be steam-atomized in the upstream set of headers at the venturi throat, and the makeup liquor would be

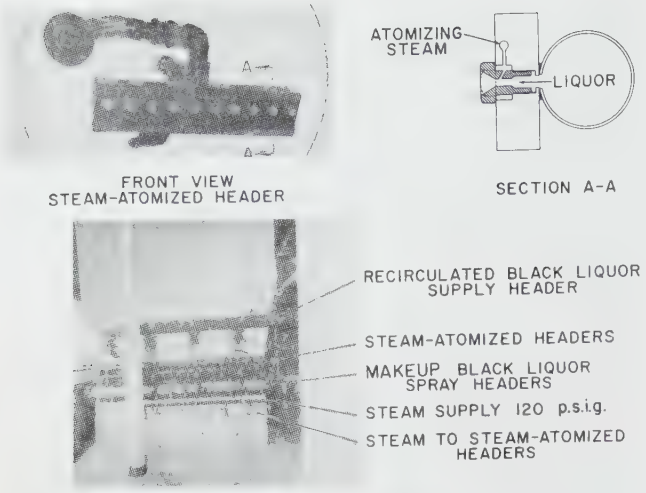

Fig. 10. The Babcock and Wilcox steam-atomized venturi scrubber

introduced into the downstream set of headers. The pilot data, shown on Fig. 9, indicated that it was possible to obtain 90% collection efficiency for 20 in. H_2O pressure drop, compared to 70% at the same pressure drop with the single header venturi. These tests also indicated that a 95% collection efficiency was attainable for a pressure drop of 30 in. H_2O, which is about 7% higher than with the single header venturi design.

Commercial Evaluation of Steam-Atomized Venturi. In March 1960, the 250-ton recovery unit at Thilmany was modified to include a dual manifold, steam-atomized venturi. This arrangement was the same as the dual manifold venturi except that the recirculated liquor was introduced through segmental steam-atomized headers as shown on Fig. 10.

The steam-atomized headers were designed with 10 steam-liquor spray jets per unit for atomizing the liquor as it entered the venturi throat. These steam-atomized, liquor-spray jets consisted of Type 316 stainless steel nozzles screwed into a carbon steel block and manifold assembly with the steam admitted to the nozzles through holes in the block. A set of conventional, splash plate headers was installed in the venturi throat section below the steam-liquor headers to distribute the low-density liquor uniformly across the width of the lower throat section.

Two series of tests were made at Thilmany to determine wheather the pilot plant predictions could be met and to demonstrate the feasibility of the new design under sustained operating conditions.

The first series was at a very low venturi differential of approximately 6 in. H_2O, and the second was at an intermediate differential of approximately 20 in. The results of these tests are shown in Table I and Fig. 9, together with a plot of the pilot data. For comparison, a curve showing the performance of the single header venturi is also included. The commercial results on the steam-atomized venturi, were comparable to the pilot unit data in the 90% efficiency range and significantly higher than those obtained with the dual manifold arrangement. At 20 in. pressure drop across the venturi throat, 70% collection efficiency was obtained with the single header venturi and 90% with the steam-atomized

Table I. Summary of Test Data
Dual Manifold Arrangement
(Buckeye Cellulose Co.)

Venturi differential, in. H_2O	29.5
Liquor/gas ratio, gal./1000 cu. ft.	9.5
Venturi throat velocity, f.p.s.[a]	230
Collection efficiency, %	90.7

[a] Based on cyclone separator outlet temperature, pressure, and density.

Table II. Summary of Test Data
Steam-Atomized Venturi
(Thilmany Pulp And Paper Co.)

Venturi differential, in. H_2O	6.3	19.5
Liquor/gas ratio, gal./1000 in. ft.	5.5	6.67
Venturi throat velocity, f.p.s.[a]	158.5	195.0
Atomizing steam flow, lb./hr.	1520	2180
Collection efficiency, %	78.1	89.0

[a] Based on cyclone separator outlet temperature, pressure, and density.

Table III. Summary of Test Data
Steam-Atomized Venturi
(D. M. Bare Paper Co.)

Venturi differential, in. H_2O	29.1
Liquor/gas ratio, gal./1000 cu. ft.	6.0
Venturi throat velocity, f.p.s.[a]	229.2
Atomizing steam flow, lb./hr.	797.0
Collection efficiency, %	93.2

[a] Based on cyclone separator outlet temperature, pressure, and density.

Table IV. Comparison Between Single Header and Steam-Atomized Venturi Scrubber

	Single header	*Steam-atomized venturi*
Venturi pressure drop, in. H_2O	30	30
Collection efficiency, %	88	94
Liquor/gas ratio, gal./1000 cu. ft.	10.0	6.0[a]
Venturi throat velocity, f.p.s.	275	230
Atomizing steam flow, lb./hr./100 tons/day		700
Cyclone exit gas temp., °F.	190	175
Availability of venturi	Same as boiler	Same as boiler

[a] Includes 45% liquor to lower set of manifolds.

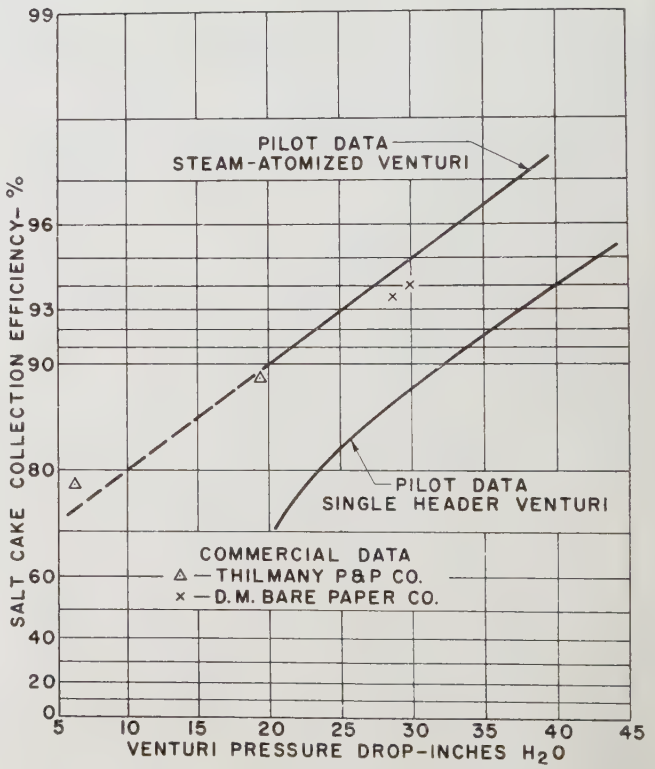

Fig. 11. Comparison of commercial and pilot data steam-atomized venturi

venturi. This increase of 20% in efficiency was achieved by using approximately 700 lb. of steam per hour per 100 tons per day of pulp production. The atomizing steam pressure is about 40 to 50 p.s.i.g. at the nozzle or 10 p.s.i.g. greater than the liquor pressure.

The steam-atomized venturi has been in continuous operation at Thilmany for approximately one year, and no operating problems have been experienced.

Since it was not possible at Thilmany to obtain a venturi differential higher than 20 in. H_2O because of physical limitations, further testing to determine the soundness of pilot-plant predictions was accomplished on the D. M. Bare Paper Co. unit, Roaring Springs, Pa. These data, shown on Table III and Fig. 11, are in reasonable agreement with the pilot data. The pilot unit predicted a collection efficiency of 95% for 30 in. pressure drop, and the commercial tests resulted in an efficiency of 94% for this pressure drop.

In comparing the performance with the single header venturi, it will be seen in Fig. 11 that the venturi scrubber collection efficiency, through the use of dual manifolds and steam atomization, has been improved by 6% at a venturi differential of 30 in. H_2O. Other areas of comparison are shown on Table IV.

Commercial Acceptance of Steam-Atomized Venturi. As a result of the successful performance and operating experiences obtained at Thilmany and D. M. Bare, three additional scrubber installations have been or will be converted to the steam-atomized venturi. In addition, there are under construction six new recovery boilers having this feature, including a 390-ton unit at Thilmany and a 125-ton unit at D. M. Bare Paper Co.

SUMMARY

It will be seen that through these developmental studies the venturi scrubber performance has been improved significantly through the incorporation of the dual header manifold and the use of steam atomization. For example:

1. The collection efficiency was increased from 88% to 94% at 30 in. H_2O pressure drop. This means that the fan horsepower remained unchanged.
2. The quantity of liquor recirculated to the venturi throat was reduced from 10.0 gal to 6.0 gal per 1000 cu. ft. of gas. This results in a savings in pump power.
3. The exit gas temperature leaving the scrubber was reduced from 190 to 175°F. This further increases the overall thermal efficiency of the boiler–scrubber combination.
4. Operating experience has indicated that the steam-atomized venturi has the same high availability that has been characteristic of the venturi scrubber since its development. This means a minimum of downtime and low maintenance costs.

LITERATURE CITED

1. Collins, T. T., Jr., Seaborne, C. R., and Anthony, A. W., Jr., *Paper Trade J.* 126, No. 3: 45–49 (Jan. 15, 1948): *Tech. Assoc. Papers* 31, 637–641 (1948).
2. Collins, T. T., Jr., and West, P. H., *Paper Trade J.* 129, No. 9: 19–22; No. 10: 25–29, (Sept. 1, 8, 1949).
3. West, P. H., *Technical Assoc. Papers* 38, 399–302 (1955).
4. *Paper Mill News* pp. 14–22 (May 18, 1957).
5. The Babcock & Wilcox Co., "Stack Loss and Venturi-Scrubber Test Sampling Instructions." (unpublished)

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Studies in the Gas and Vapor Permeability of Plastic Films and Coated Papers

Part VII. The Permeation of Organic Vapors

C. E. ROGERS, V. STANNETT and M. SZWARC

Certain aspects of the permeability, *P*, diffusivity, *D*, and solubility, *S* of organic vapors in polymers, especially polyethylene, have been summarized to illustrate the dependence of these phenomena on the characteristics of the polymer–penetrant system. The following factors affecting *P*, *D*, *S* are discussed with reference to results of recent investigations: the chemical nature of the penetrant and the polymer; the morphology of the polymer including cross-linking and crystallinity; the concentration, size, and shape of the penetrant; the temperature; and the nature of composite film barriers. Relationships are presented which describe the dependence of *P*, *D*, *S* on the penetrant concentration, activity, shape, and size. An expression based on polymer solution theory, as modified to account for the cross-linking action of impenetrable polymer crystallites, allows a calculation to be made of the penetrant–polymer interaction parameter and the molecular weight of the amorphous material between crystallites. The mechanism of the diffusion and sorption processes are discussed in terms of the segmental motions required for passage of a penetrant molecule through the polymer structure. Under certain conditions of temperature and concentration the plasticizing and swelling action of an imbibed penetrant drastically alters the physical nature of the polymer medium. The concurrent permeation, diffusion, and sorption behavior in these systems often is anomalous, but may be of some practical significance for the selective permeation of vapors.

NOMENCLATURE

p = vapor pressure, cm. Hg
p^0 = saturated vapor pressure, cm. Hg
a_1 = vapor activity = p/p^0
$\overline{V}_1$ = molar volume of condensed vapor, cc./mole
ρ = whole polymer density, g./cc.
ρ_a = density of amorphous regions in polymer, g./cc.
M_c = molecular weight of amorphous regions between crystallites
c = concentration of sorbed penetrant in polymer
c_w = concentration of sorbed penetrant in whole polymer, g. vapor/g. polymer

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c_a = concentration of sorbed penetrant in amorphous regions of polymer, g. vapor/g. amorphous polymer

c_1 and c_2 = concentration of gas or vapor at high- and low-pressure surface, respectively.

V_{1a}, V_{2a} = volume fractions of sorbed penetrant and amorphous polymer, respectively, in the penetrant-amorphous polymer mixture

Q/Q_e = fractional weight gain, or loss, during the sorption or desorption process

μ_1 = Flory-Huggins solution theory interaction parameter

χ_1 = "apparent" value of μ in a semicrystalline polymer

P = permeability coefficient, cc. (S.T.P.)/mm./cm.^{2}/sec./cm. Hg

D = diffusion coefficient, cm.^{2}/sec.

S = solubility coefficient, g/g/cm. Hg

$P(0)$ = limiting permeability coefficient at zero concentration

$D(0)$ = limiting diffusion coefficient at zero concentration

$S(0)$ = limiting solubility coefficient at zero concentration

$\bar{D}$ = integral or mean value of D over a range of concentration

σ, γ = parameters characterizing the concentration dependence of S and D, respectively.

α = parameter characterizing the activity dependence of D

$\mathcal{S}(0)$ = limiting value of $S(0)$ at zero molar penetrant volume

G = parameter characterizing the dependence of $S(0)$ on $\bar{V}_1$

P_0 = pre-exponential factor in the Arrhenius temperature equation for permeation; the value of P at "infinite" temperature

D_0 = pre-exponential factor in the Arrhenius temperature equation for diffusion; the value of D at "infinite" temperature

S_0 = pre-exponential factor in the Arrhenius temperature equation for solubility; the value of S at "infinite" temperature

E_p = activation energy for permeation, kcal/mole

E_D = activation energy for diffusion, kcal/mole

ΔH = heat of solution of penetrant in polymer, kcal/mole

INTRODUCTION

Previous papers in this series have dealt primarily with the transmission of the permanent gases and water vapor through plastic films and coated papers. The sorption and diffusion of organic vapors is of considerable practical importance from the point of view of odor and flavor retention by packaging materials, and in the contamination of containers by their previous contents. For example, in the case of long storage of a mixed liquid, one component may diffuse away faster than another, thus changing the composition. The sorption and diffusion behavior of organic vapors is complex and has been the subject of extensive research. This paper summarizes the work carried out with organic vapors in the authors' laboratories, together with illustrations from other recent work in this field.

In ideal gas–polymer systems, both the solubility coefficient, S, and the diffusion coefficient, D, are constant at any given temperature so that the permeability, $P \equiv DS$, is also a constant (*1*). With organic vapors these coefficients are usually some function of concentration and perhaps of the time scale of the experiment and the history of the polymer sample.

The ideal gases interact little, if at all, with polymers, and the amount sorbed is sufficiently small so that the polymer matrix does not undergo any swelling strain or other rearrangement during the permeation process. The situation becomes more complex and deviations from ideality more extreme as the vapor becomes less ideal and as the penetrant–polymer interaction increases. The higher concentrations of sorbed vapor swell and plasticize the polymer, leading to increased mobilities of both polymer segments and penetrant molecules. Indeed, in the presence of a diffusing solvent, the morphology of the polymer may be drastically altered owing to enhanced stress relaxation and orientation, thereby strongly affecting the concurrent diffusion, sorption and permeation behavior.

The form and magnitude of these effects are determined by the chemical and physical properties of the penetrant and polymer, and by the external conditions of the system such as temperature and pressure.

Certain aspects of the sorption, diffusion, and permeation of organic vapors in polymers, especially polyethylene, will be discussed to illustrate the dependence of these phenomena on the characteristics of the system.

THE NATURE OF THE PENETRANT AND POLYMER

From thermodynamic considerations, it is predicted that the more easily condensible vapors would be more soluble in a given polymer, and this is borne out by the approximately linear relationship between the logarithm of the solubility and the boiling point of the gas or vapor (*1*).

The solubility of a vapor in a polymer depends on their mutual compatibility and, in general, the principle of "like dissolves like" is applicable. The permeability, in turn, also increases with similarity in chemical structure between the penetrant and the polymer. For example, the permeation rate through polyethylene is lowest with strongly polar materials and greatest with hydrocarbons in the following general order (*2*): alcohols, acids, nitro-derivatives, aldehydes and ketones, esters, ethers, hydrocarbons and halogenated hydrocarbons; some typical data (*3*) are given in Table I.

It may be observed that in almost all cases the liquid-permeability rate exceeds the vapor-permeability rate. This phenomenon probably is due to the effect of the intervening static air space between the lower surface of the membrane and the liquid surface in the vapor-meter cup used to obtain these data (*32*). A vapor-pressure gradient is established across this air space so that the effective vapor pressure at the film surface,

Table I. Vapor and Liquid Permeabilities (*3*) of Polyethylene at 80°F.

	Permeation rate, g/24 hr./100 in.^{2}/mil thickness		
Compound	*Density ≈ 0.96 g./cc. Liquid*	*Vapor*	*Density ≈ 0.92 g./cc. Vapor*
Amyl alcohol	0.268	0.353	...
Ethylene glycol	0.873	0.669	...
Acetic acid	0.993	0.940	...
Aniline	1.27	1.09	3.96
Ethyl alcohol	1.77	1.26	...
Oil of peppermint	1.75	1.36	3.68
Butyric acid	3.61	1.54	...
Benzaldehyde	3.92	2.47	9.67
Methyl salicylate	3.92	3.39	...
Methyl ethyl ketone	8.05	4.12	...
Amyl acetate	6.75	5.54	...
Diethyl ketone	7.67	7.67	...
Ethyl valerate	9.69	8.11	...
Butyraldehyde	12.6	9.86	...
Ethyl acetate	13.7	13.7	18.2
n-Butyl ether	34.3	19.3	...
Butyl iodide	113.	52.0	...
Heptane	69.7	54.0	...
Butyl chloride	86.05	69.6	...
Butyl bromide	113.	76.15	...
Benzene	...	103.	729.
Carbon tetrachloride	...	120.	580.

Table II. The Permeability (4) of Selected Polymers to Ethylene Oxide and Methyl Bromide

Polymer	Permeability, P, cc. STP/mm./cm.²/sec./cm. Hg	
	Ethylene oxide T = 0°C. p = 330 mm. Hg	Methyl bromide T = 60°C. p = 395 mm. Hg
Polyvinyl alcohol	0.0002×10^{-8}	...
Polyester (Mylar)	...	0.008×10^{-8}
Polyvinylidene chloride (Saran)	.	0.08
Polytrifluorochloroethylene (Kel-F)	0.12	0.46
Cellulose acetate, plasticized	140.	0.68
Polyamide (nylon 6)	...	0.84
Rubber hydrochloride (Pliofilm)	73.	3.4
Polyethylene, 0.92 g./cc.	4.9	47.2
Ethyl cellulose, plasticized	42.	...

which determines the permeation rate, is different for the two cases. When vapor-permeability rates are determined in the absence of interfering gases (*35*), or when the air–vapor mixture is circulated over the film surface (*1*), the liquid-permeability and saturated vapor-permeability are identical (*8*, *35*). The additional resistance to vapor flow caused by the presence of other gases has an important bearing on the practical application of permeation data obtained in the absence of such gases.

The exact sequence of increasing permeability of a given vapor in a series of polymers depends not only upon the interactions between the penetrant and the polymer but also to some extent on the nature of the polymer itself. Any factor which tends to make the polymer-chain segments less mobile, or pack more closely, will decrease the permeation rate. Other factors being equal, the permeation rate can be expected to decrease as the symmetry and cohesive energy density of the polymer increases. In Table II, data are given for the permeation of two organic vapors through several polymers of quite different structure and polarity (*4*).

MORPHOLOGY OF THE POLYMER

Cross-linking

Studies of the effect of cross-linking on the gas and vapor permeability of rubbers (*5*, *6*) have shown that the diffusion coefficients decrease with increasing degrees of cross-linking while the solubility is relatively unaffected, except at very high degrees of cross-linking or of swelling. The introduction of cross-links decreases the diffusion coefficient at zero concentration, $D(0)$, and makes the diffusion coefficient more dependent on concentration.

The same results are obtained in the case of methyl bromide and polyethylene (*7*); irradiation (10^8 roentgen) and subsequent cross-linking does not affect the solubility but does decrease the permeability to about half that of the unirradiated polymer. For larger penetrants (*8*), which may be better solvents and swelling agents than methyl bromide, both the solubility and diffusion are increased slightly at low temperatures due to the small decrease in polymer crystallinity caused by the irradiation process. At higher temperatures, S and D are both decreased since the cross-links impose restraints on the amount of swelling, as well as decreasing the polymer segment mobility.

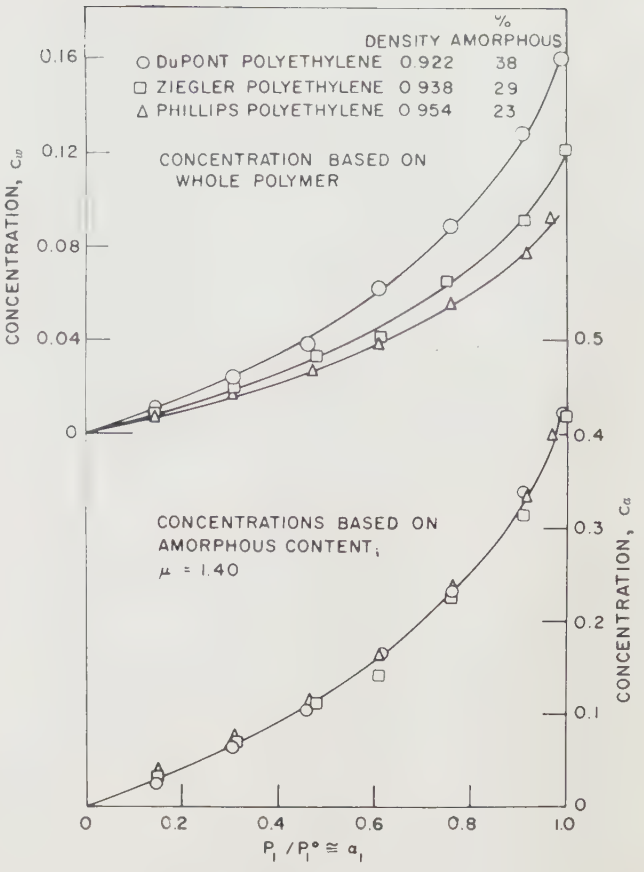
% DENSITY AMORPHOUS
○ DuPONT POLYETHYLENE 0.922 38
□ ZIEGLER POLYETHYLENE 0.938 29
△ PHILLIPS POLYETHYLENE 0.954 23
CONCENTRATION BASED ON WHOLE POLYMER
CONCENTRATIONS BASED ON AMORPHOUS CONTENT, μ = 1.40
CONCENTRATION, C_p
CONCENTRATION, C_a
$P_1 / P_1^\circ \cong a_1$

Fig. 1. Sorption isotherms for polyethylenes and methyl bromide, at 0°C.

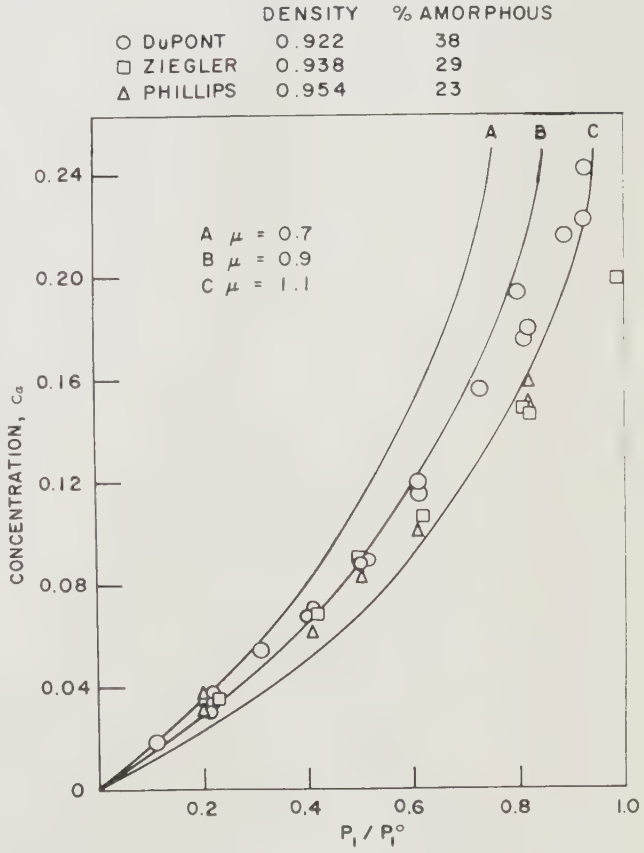

Fig. 2. Sorption isotherms for polyethylenes and n-hexane, at 0°C., calculated on the basis of amorphous content

Since the energy requirements for the overall permeation process are only slightly affected by increased cross-linking and penetrant size, the major contribution to the drop in permeability must be due to a decrease in the temperature-independent factor $P_0 \equiv D_0S_0$. When the penetrant size is small and the amount of swelling is negligible, the decrease of P_0 is due to a decreased value of D_0, related to decrease in the entropy of activation for the diffusion process. The probability of the polymer chains moving away from some central point, thus forming a hole for passage of the penetrant, will obviously be diminished if the chain segments are tied together at intervals by cross-links. A simultaneous decrease in the factor S_0, for more bulky penetrants, is similarly related to the decreased probability that the polymer chains will move so as to form a sufficient volume to contain the diffusing penetrant molecule.

Crystallinity

The sorption isotherms for a given vapor differ with different-density materials (*9–11*); typical isotherms are shown in Figs. 1 and 2. It can be assumed that sorption occurs only in the amorphous regions of the polymer. In the lower curve of Fig. 1 and in Fig. 2, the previous isotherms have been recalculated on the basis of the amorphous content of the three polyethylene samples. The concentration is given as grams vapor/grams polymer, c_w, and grams vapor/grams amorphous polymer, c_a.

In the case of methyl bromide, a poor solvent, the amorphous-polymer isotherms lead to a single value of 1.40 for μ, the Flory-Huggins interaction parameter (*33*), for the three samples, whereas sorptions based on the whole polymer lead to different μ values for each polymer. The range of μ values for the whole-polymer sorption is unlikely since, chemically, the polymers are largely identical.

In the case of *n*-hexane, a good solvent, no one value of μ adequately represents the isotherms, regardless of whether the sorption is based on the whole polymer or on the amorphous content. For the amorphous-polymer sorption, μ varies from 0.7 to 1.2 for all three samples and any attempt to fit the amorphous sorption isotherms with any single value of μ is unsatisfactory, as is shown in Fig. 2. Furthermore, the μ values are too high for a good solvent.

Since good solvents, such as *n*-hexane, swell the polymer considerably, it is necessary to modify the usual Flory-Huggins equation by the addition of a term for the entropy of elasticity, tending to restrain the swelling of the polymer, which is "cross-linked" by the crystallites. This leads to the expression (*12*):

$$\ln p/p^0 \cong \ln a_1 = \ln V_{1a} + V_{2a} + \mu_1 V_{2a}^2 + \frac{\rho_a \bar{V}_1}{M_c} V_{2a}^{1/3}$$

$$= \ln V_{1a} + V_{2a} + \left[\mu_1 + \frac{\rho_a \bar{V}_1}{M_c} V_{2a}^{-5/3}\right] V_{2a}^2$$

$$= \ln V_{1a} + V_{2a} + \chi_1 V_{2a}^2 \qquad (1)$$

The linear plots of χ_1 versus $V_{2a}^{-5/3}$ serve to determine simultaneous values of μ_1 and M_c; μ_1 from the intercept and M_c from the slope. The values of μ_1 and M_c determined by this method are given in Table III.

It is not to be expected that the value of μ_1 as calculated by this method will *necessarily* be identical with that calculated from data obtained from homogeneous solutions of solvent and polymer. The relative number of solvent-solvent, polymer–polymer, and solvent–polymer contacts is very different in the case of initially sorbed vapor as opposed to the saturated solution; the magnitudes of the heat and entropy contribution will possibly be quite different in the two instances. The values of μ_1 by this method are generally lower than those found from homogeneous solution, but still follow the same trends predicted by theories of polymer solutions. For example, μ_1 decreases as the amount of

Table III. Solubility Parameters, Molecular Weight between Crystallites, and Interaction Parameter for Polyethylene

Polymer density, g./cc.	*Vapor*	*Key*	*Temp., °C.*	S(0), *g./g./cm. Hg*	σ, *g./g.*	M_c	μ_1
0.919	Methyl bromide	...	0	1.1×10^{-2}	5.9	...	...
		...	30	0.4	4.4	...	...
0.922	Isobutylene	B1	−8	0.49	8.6	...	...
		B2	0	0.40	7.5	...	...
		B3	30	0.21	9.3	...	...
	n-Pentane	C	25	1.1	8.1	250	0.22
	n-Hexane	...	0	11.	6.5	160	0.01
		D2	25	3.9	6.4	150	−0.063
		...	30	3.6	5.3	160	−0.16
	n-Heptane	E	25	11.	6.3	220	0.096
	n-Octane	F	25	46.	4.4	140	−0.31
	Cyclohexane	G	25	8.3	5.3	320	0.31
	Benzene	H1	0	19.	7.5	...	...
		H2	25	7.5	5.5	230	0.50
	Toluene	I	25	24.	5.5	230	0.46
	Ethyl benzene	J	25	78.	4.4	210	0.41
	p-Xylene	K	25	98.	3.9	160	−0.054
	Chloroform	...	25	5.9	3.5	290	0.64
	Carbon tetrachloride	...	25	13.	2.7	160	0.10
	Methyl bromide	...	0	1.2	5.5	∞	1.40
		...	25	0.57	5.8	∞	...
		...	30	0.49	6.5	∞	1.20
	Ethyl bromide	...	25	2.	4.6	∞	1.02
0.938	Methyl bromide	...	0	0.84	6.5	∞	1.40
			30	0.33	7.0	∞	1.25
	n-Hexane	...	0	8.7	5.4	82	−0.56
			30	2.3	6.3	100	−0.37
0.954	Methyl bromide	...	0	0.74	7.0	∞	1.40
			30	0.23	9.9	∞	1.35
	n-Hexane	...	0	6.7	7.3	82	−0.52
			30	2.0	6.8	100	−0.47

polymer-chain branching decreases, in agreement with the behavior predicted by Doty, *et al* (*13*), and shown experimentally by Richards (*14*), and by Muthana and Mark (*15*).

The decrease in M_c with polymer density correlates with the decrease in the amorphous content of these samples. The value of M_c for the more highly branched polyethylene (ρ = 0.922 g./cc.) agrees well with the M_c predicted by consideration of the chain length between branch points as determined by infrared measurements of the CH_3/C ratio, if it is assumed that branch points and chain ends are excluded from the crystallites.

The low value of M_c for the higher density polyethylenes leads to a correspondingly low value for the crystallite size in these polymers. The values for the crystallite sizes of the polyethylenes do not agree well with the results of X-ray determination of crystallite size. It is quite probable that sorption of solvents in polyethylene may induce structural changes not unlike local melting, to an extent that depends on penetrant size and solvent power, and on the degree of order of the amorphous regions, quasi-crystallinity increasing with the X-ray determined "true" crystallinity. If such is the case, then the penetrating solvent would break up large imperfect crystallites into many smaller crystallites during the sorption and swelling process. This would alter the crystallite size but would not substantially change the overall percentage of crystallinity.

It should be emphasized that the foregoing treatment is oversimplified and is presented only to give an indication of the type of considerations involved.

At the present time, there does not appear to be any simple relationship between the initial polymer density and P (and D) for organic vapors which swell the polymer. Variations in polymer density in the absence of vapor are due to structural differences, such as chain branching, and to the history of the sample, such as the temperature and pressure during the crystallization process. The amorphous regions, through which permeation probably takes place, are somewhat different from sample-to-sample. The crystalline regions may affect the permeability by acting both as impermeable fillers and as "cross-linking" areas. The presence of a solvent, distributed in a concentration gradient through the film, undoubtedly disrupts the initial local configurations of the crystalline–amorphous regions. The effective density and the local configurations in the polymer–solvent mixture will then vary in a nonlinear fashion from one surface of the membrane to the other. The diffusion process in the resultant nonhomogenous medium is quite complex.

In the case of gases which do not swell the polymer to any significant extent, the permeation process is not as complicated. Michaels and Parker (*34*) have discussed in considerable detail the effects of crystallinity on gas permeability.

The decrease in the Arrhenius parameter $P_0 = D_0S_0$, with increase in polymer crystallinity (*10*) at a given temperature is easily understood in terms of the preceding discussion of the decrease of these same parameters with increasing cross-linking of a polymer. However, the crystalline "cross-links" are temperature-dependent since crystallinity decreases with increasing temperature, so that the entropies of activation for diffusion and sorption are, in a sense, temperature-dependent also.

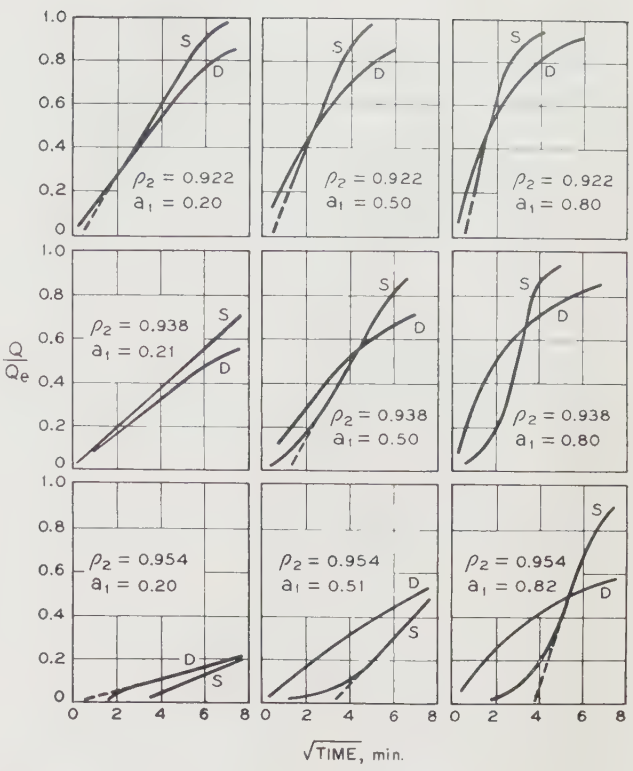

Fig. 3. Sorption–desorption curves for n-hexane at three vapor activities (a_1) in different-density polyethylenes, at 0°C.

S = Sorption; D = Desorption

It should be mentioned that detailed analysis of the concentration and temperature dependence of S, D, and P for vapors in ethyl cellulose (*16*) strongly suggests that there are pre-existing cavities, or voids, in the polymer able to accommodate certain penetrant molecules. There are indications that similar "voids" are also present in other polymers, such as polyethylene (*17*); the exact nature of these cavities and their effect on permeation have not yet been established.

ANOMALOUS SORPTION AND DIFFUSION

Complicating features arising in diffusion in the more rigid and internally viscous polymers are due to time or history effects. Polymers at temperatures below the glass temperature and those polymers with long relaxation times usually exhibit anomalous non-Fickian diffusion and sorption behavior. Discussions of these phenomena and many references to the literature are to be found in recent publications (*18–20*).

It might be expected that highly crystalline polymers would, in general, be included in this class of non-Fickian systems owing to the probable change of crystalline content and order in the presence of diffusing solvents.

Figure 3 shows the sorption–desorption curves for *n*-hexane in different-density polyethylenes (*21*). Since Fick's law predicts a linear initial slope (*19*) for sorption data, as plotted in Fig. 3, it is apparent that the sorption process becomes progressively less ideal as the polymer density increases. Further studies show that the deviations from ideality increase as the initial order of the polymer, the solvent power and the concentration of the penetrant all increase. Since the diffusion behavior in these time-dependent systems is definitely non-Fickian,

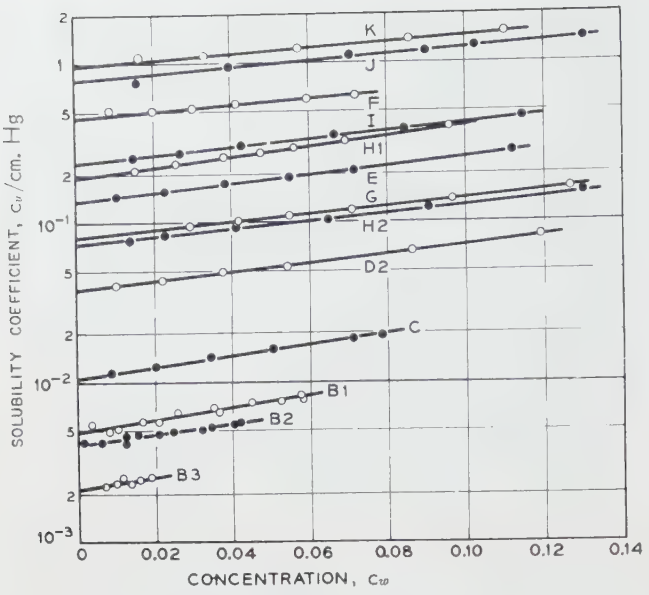

Fig. 4. Solubility coefficients vs. concentration; polyethylene density = 0.922 g./cc.

Key to symbols given in Table III.

in the usual sense, any calculation of the diffusion coefficient based on Fick's law will be only approximate. Values of $\bar{D}$ may be calculated using the intermediate linear portion of the sorption curve on the assumption that this is the rate of sorption that will be present in the final steady state of permeation.

THE EFFECTS OF PENETRANT CONCENTRATION ON *S*, *D*, AND *P*

The solubility coefficient often is essentially constant at low vapor activities for the more volatile vapors. At higher vapor activities, or with more condensable vapors, the sorption may deviate significantly from the behavior predicted by Henry's law.

In an investigation of the sorption of hydrocarbon vapors in different-density polyethylenes (*21*), it was found that the data could be represented by a linear function of the logarithm of the solubility coefficient ($S = c/p$) versus the concentration, c of the sorbed vapor; typical plots are shown in Fig. 4.

These isotherms may be represented by the expression:

$$S = S(0) \exp(\sigma c) \qquad (2)$$

where $S(0)$ is the solubility coefficient from the intercept at $c = 0$, and is a measure of the initial sorption of unassociated penetrant into a polymer matrix which is undisturbed by stresses imposed by the sorption process. The parameter σ is a constant characterizing the concentration dependence of the solubility coefficient. When the solubility coefficient is not concentration dependent $\sigma = 0$, and the solubility is a constant as predicted by Henry's law.

Values of $S(0)$ and σ for the sorption of organic vapors in different polyethylenes are given in Table III. The above expression for the concentration dependence of the solubility coefficient (which is directly related to the reciprocal of the activity coefficient) also adequately represents data for many other polymer–penetrant systems (*22*) where there are no specific penetrant–polymer interactions such as solvation or other penetrant localization.

By using relations derived by Zimm and Lundberg (*23*), involving the rate of change of the activity coefficient with activity, it has been shown (*21*) that there is considerable clustering of penetrant molecules within the polymer matrix of polyethylene during the sorption process. The magnitude of this clustering effect increases as the solvent power of the penetrant decreases and as the sorbed concentration increases. The clustering in the higher density polyethylenes is initially lower than that in the lower density polymer but shows a more marked increase with increase in concentration. The detailed dependence of clustering on the above factors and on the size and shape of the penetrant suggests that the sorption of vapors by polyethylene may occur in two successive modes. The initial sorption is random mixing of solvent and amorphous polymer up to a critical-volume fraction, which is dependent on the degree of crystallinity. Subsequent sorption of vapor past this critical-volume fraction results in formation primarily of solvent clusters within the polymer matrix.

As stated previously, it has been found that in most organic vapor–polymer systems the diffusion process is strongly dependent upon the concentration of the penetrant in the polymer. In such cases, a diffusion coefficient experimentally deduced under nonsteady or steady-state conditions of penetrant flux is usually some kind of mean or integral value $\bar{D}$, over the range of concentration involved:

$$\bar{D} = [1/(c_1 - c_2)] \int_{c_2}^{c_1} D \, dc \qquad (3)$$

Estimates of the integral diffusion coefficient for organic vapors in polyethylene were determined (*21*) by three different methods; viz., the time-lag method, the rate of sorption–desorption and the quotient of the permeability constant and the equilibrium solubility coefficient. These methods assume that the diffusion process at least approximates to Fick's law without the complications due to swelling and time-dependent relaxation phenomena. Satisfactory agreement between the three methods was obtained, within experimental error (see Fig. 5). For *n*-hexane the $\bar{D}$ values calculated from steady-state data ($\bar{D} = P/S$) initially agree with the values calculated by the nonsteady-state methods, but have a greater dependence on activity (curve g', Fig. 5). If time effects are present in the sorption–diffusion process, it is expected that diffusion coefficients measured by transient-state methods will be smaller than those measured by equilibrium methods, as was found by Park (*24*) for diffusion in polystyrene. As shown for the diffusion of *n*-hexane in polyethylene, the difference between the two estimates of the diffusion coefficient approaches zero as the penetrant concentration goes to zero.

The diffusion coefficients for the better solvents (isobutylene, benzene, and *n*-hexane) are exponential functions of the vapor activity, Fig. 5; while the same data plotted versus concentration give curves concave to the concentration axis. Meares (*20*) has noticed a similar curvature for the diffusion of allyl chloride in polyvinyl acetate when log D is plotted over a wide range of concentration.

The dependence of the logarithm of $\bar{D}$ on vapor activity suggests that the data may be represented by:

$$\bar{D} = D(0) \exp(\alpha a_1) \qquad (4)$$

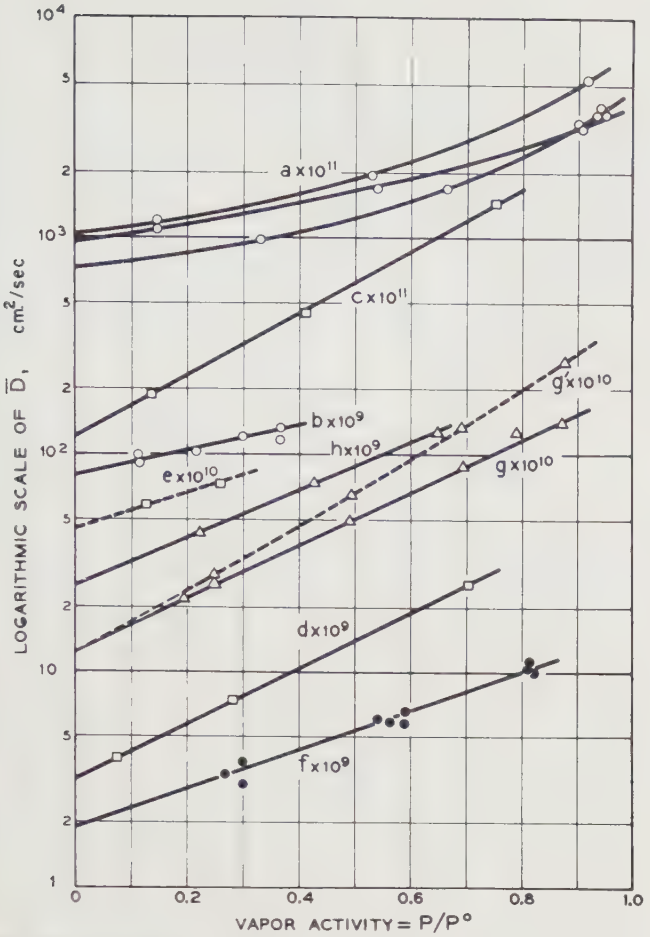

Fig. 5. ***Diffusion coefficients vs. vapor activity; polyethylene density = 0.922 g./cc.***

Key to symbols given in Table IV.

where $D(0)$ is the diffusion coefficient at zero activity, pressure, and concentration, while α is a constant for a penetrant–polymer system at a given temperature.

From equation (2), representing the dependence of sorbed vapor concentration on pressure, it follows that:

$$\bar{D} = D(0) \exp\ [\gamma c/\exp(\sigma c)] \qquad (5)$$

with:

$$\gamma = \alpha/p^0 S(0) \qquad (6)$$

Expanding the exponential involving σc and neglecting the higher power terms (valid for small σc) yields:

$$\bar{D} = D(0) \exp\ [\gamma c/(1 + \sigma c)] \qquad (7)$$

When the gas or vapor obeys Henry's law, $\sigma = 0$ (S then being a constant independent of c or p), and the expression becomes:

$$\bar{D} = D(0) \exp(\gamma c) \qquad (8)$$

which is an equation often reported for the diffusion of ideal gases and vapors at low concentrations (*6, 18–20, 26–28*).

The permeability constant follows from equations (2) and (4) as:

$$P = P(0) \exp[\alpha a_1 + \sigma c] \qquad (9)$$

with

$$P(0) = D(0)S(0) \qquad (10)$$

Since the sorbed vapor concentration increases rapidly with increasing pressure, a plot of log P versus vapor activity becomes progressively nonlinear (see Fig. 6). When the solubility of the vapor approximates to Henry's law or, in general, at sufficiently low vapor activities, equation (9) reduces to a linear dependence of P on vapor pressure, as was found previously by Cutler, Kaplan, McLaren, and Mark (*25*):

$$P = P(0) + Kp \qquad (11)$$

From the observed activity dependence of $\bar{D}$ it appears that the plasticizing effect of the sorbed vapor overcomes any possible effect of increasing penetrant-cluster size. The plasticizing action tending to increase $\bar{D}$ is greater, by a factor $\exp\{\alpha a_1\}$, than any clustering effect tending to decrease $\bar{D}$. It may be assumed that the mobility of the penetrant clusters is so low that essentially no migration of clusters per se occurs within the polymer, so that the activity dependence of $\bar{D}$ is that for the diffusion of single molecules through a medium by a process of condensation and evaporation; i.e., spatial movement of penetrant occurs by migration of single molecules from site-to-site within the polymer–penetrant mixture.

Values of α, γ, $D(0)$, and $P(0)$ for the diffusion and permeation of vapors in polyethylene are presented in Table IV. The parameter γ has been shown by Long and co-workers (*26, 27*) to be a measure of the loosening of the polymer structure, related to the change in viscosity or plasticity which occurs as the penetrant concentration increases. The data of Table IV show two opposite trends of γ with the solvent power of the penetrant as measured by the relative value of the Flory-Huggins interaction free energy parameter, μ. The value of μ decreases from poor to good solvents in the order: methyl bromide, benzene, hexane; in this same sequence the value of γ at 0°C increases. This indicates that the more strongly interacting penetrant has the more pressure- and concentration-dependent diffusion and permeation behavior at a given temperature. This relationship does not carry over when the

Table IV. Diffusion and Permeation Parameters for Polyethylene

Polymer density, g./cc.	*Vapor*	*Key*	*Temp., °C.*	*α*	*γ, g./g.*	D(0), *cm.²/sec.*	P(0), *cc. STP/mm. cm.²/sec./cm. Hg*
0.919	Methyl bromide	a	0	0.9	12	7.3–10 × 10^{-9}	1.3 × 10^{-8}
		b	30	1.3	14	83.	7.9
0.922	Isobutylene	c	−8	3.3	92	1.25	0.23
		d	0	2.9	70	3.1	0.45
		e	30	1.9	35	47.	3.6
	Benzene	f	0	2.0	45	1.9	9.4
	n-Hexane	g	0	2.8	53	1.2	2.8
		h	30	2.5	36	25.	22.
0.938	Methyl bromide	...	0	2.9	53	29.	...
	n-Hexane	...	0	4.6	110	0.15	...
0.954	Methyl bromide	...	0	3.3	70	1.4	...
	n-Hexane	...	0	4.7	150	0.075	...

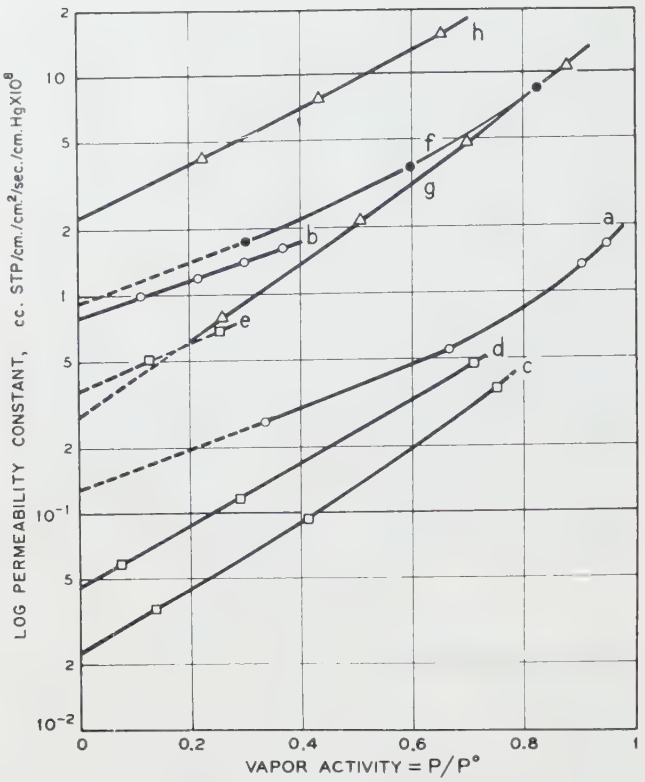

Fig. 6. Permeability constants vs. vapor activity; polyethylene density = 0.922 g./cc.

Key to symbols given in Table IV.

temperature dependency is considered, since both μ and γ decrease with an increase in temperature.

For an analogous phenomenon occurring in the diffusion of C_4 and C_5 paraffins in rubber, Barrer and Aitken (*6*) suggested that the internal mobility of the polymer chains increased so much at the higher temperatures that small increments of concentration of vapor no longer led to much additional plasticizing of polymer. At lower temperatures, the natural mobility of polymer chains was much less, and so plasticizing by penetrant molecules produced a greater effect.

A similar explanation probably applies to polyethylene since the diffusion of any given vapor becomes more concentration-dependent as the degree of crystallinity of the polymer increases. The increase in crystalline content undoubtedly decreases the natural mobility of the remaining amorphous chain segments, and so the plasticizing effect of small increments of penetrant increases.

It should be noted that the presence of water vapor has an accelerating effect on the concurrent sorption and diffusion of organic vapors in those polymers which also sorb significant amounts of water vapor (*28*). It is reasonable to assume that the smaller, generally more mobile, water molecules diffuse in advance of the organic molecules and plasticize the water-sensitive polymer, thus facilitating the diffusion of the organic vapor.

SIZE AND SHAPE OF THE PENETRANT

The magnitude of the solubility coefficient, $S(0)$, is dependent on the volume and shape of the penetrant molecule as shown by the plot of log $S(0)$ versus the molar volume $\bar{V}$ of the condensed vapor (*21*) (see Fig. 7).

Two general classes, characterized by different molecular configurations, may be distinguished: the

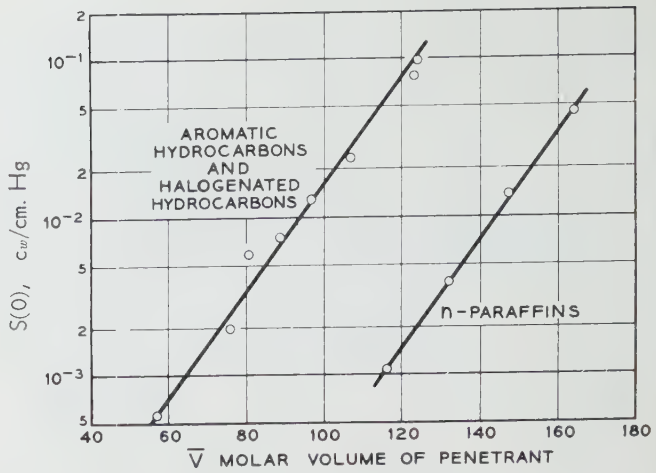

Fig. 7. Effect of molar volume of penetrant on S(0); polyethylene density = 0.922 g./cc. at 25°C.

straight-chain paraffins in one group, and the aromatic and halogenated compounds in the other. Approximate linear relationships may be represented by the equation:

$$S(0) = \mathbb{S}(0) \exp(G\bar{V}_1) \qquad (12)$$

where $\mathbb{S}(0)$ is the intrinsic solubility coefficient for a penetrant of zero molar volume and G is a constant independent of the configuration of the vapor molecules. It appears probable that $\mathbb{S}(0)$ is dependent mainly on the shape of the penetrant molecule, while G is dependent on the nature of the polymer itself. For the data of Fig. 7, G is 0.0785 mole per cc. and $\mathbb{S}(0)$ is 1.2×10^{-7} g. per g. per cm. Hg for the normal paraffins and 6.4×10^{-6} g. per g. per cm. Hg for the aromatic and halogen compounds.

The diffusion coefficients at zero vapor concentration, D(0), generally decrease as the volume and the shape of the penetrant molecule increase. From the data of Table V, for the diffusion of C_4 and C_5 paraffins in polyisobutylene (*26*), rubber (*6*), and ethyl cellulose (*16*), it is seen that branching has a much greater effect on the diffusion coefficient than does molecular size. Addition of a methyl side-chain to a given paraffin reduces the value of $D(0)$ by a larger amount than does an increase of chain length by one carbon atom. This suggests that diffusion occurs preferentially along the direction of greatest length of the penetrant molecule. The values of D(0) for C_4 and C_5 paraffins have been linearly correlated (*6*) with the product $l_1 l_2$, where l_1 is the minimum molecular dimension of the diffusing molecule and l_2 is the next smallest dimension perpendicular to l_1.

Kokes and Long (*27*) have found a marked dependence on penetrant size and shape for diffusion in polyvinyl acetate at temperatures above the polymer glass temperature, and Park (*29*) has found a similar dependence for the diffusion of halogenated hydrocarbons

Table V. Dependence of D(0) on the Size and Shape of Penetrant

Vapor	$D(0)$, cm.²/sec. Polyisobutylene, 35°C.	Ethyl cellulose, 50.1°C.	Rubber, 50°C.
Propane	4.81×10^{-9}	... $\times 10^{-9}$	... $\times 10^{-9}$
n-Butane	3.29	11.8	720
Isobutane	1.46	2.1	470
n-Pentane	2.59	5.6	680
Isopentane	1.34	2.3	440
Neopentane	0.60	2.2	260

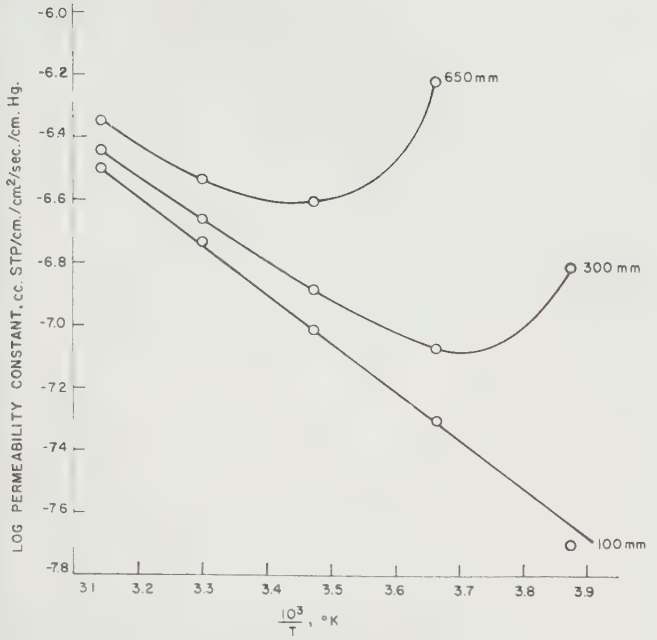

Fig. 8. Permeability isobars for methyl bromide and polyethylene (0.922 g./cc.)

in polystyrene at temperatures well below the glass temperature of the polymer.

These investigations indicate that the value of $D(0)$ decreases exponentially both as the overall molecular volume increases and as the smallest cross-sectional molecular area increases. On this basis, it may be concluded (*29*) that the probability of a diffusing molecule moving from one position in the polymer to another is proportional to the probability of finding a gap between the polymer chains wide enough to pass the penetrant as well as to the probability of finding a hole in the polymer matrix large enough to accommodate it. For larger molecules to pass through, larger segments of the polymer must move for hole formation leading to increased E_D and decreased D_0. However, other factors such as chain flexibility and the segmental chain length involved per unit diffusion step must also affect the ease of diffusion so that the stated dependence on hole size and volume can only be part of the actual conditions governing diffusion.

As mentioned before, the value of S(0) *increases* exponentially with increase in overall molecular volume and also, apparently, increases as the cross-sectional area of the penetrant increases. As a consequence of this nearly compensating dependence of $D(0)$ and $S(0)$ on penetrant size and shape, the zero-concentration permeability coefficient, $P(0) \equiv D(0)S(0)$, is much less dependent on the size and shape of the penetrant than are either $D(0)$ or $S(0)$ individually.

EFFECT OF TEMPERATURE

In general, the temperature dependences of P, D, and S are given by the Arrhenius relationships:

$$D = D_0 \exp(-E_D/RT) \quad (13)$$

$$S = S_0 \exp(-\Delta H/RT) \quad (14)$$

$$P = P_0 \exp(-E_P/RT) \quad (15)$$

where ΔH is the heat of solution, E_D and $E_P = \Delta H + E_D$ are the activation energies for the diffusion and permeation processes, and $P_0 = D_0S_0$.

For easily condensable vapors, ΔH is usually negative due to the contribution of the heat of condensation, and thus S decreases with increasing temperature. To a

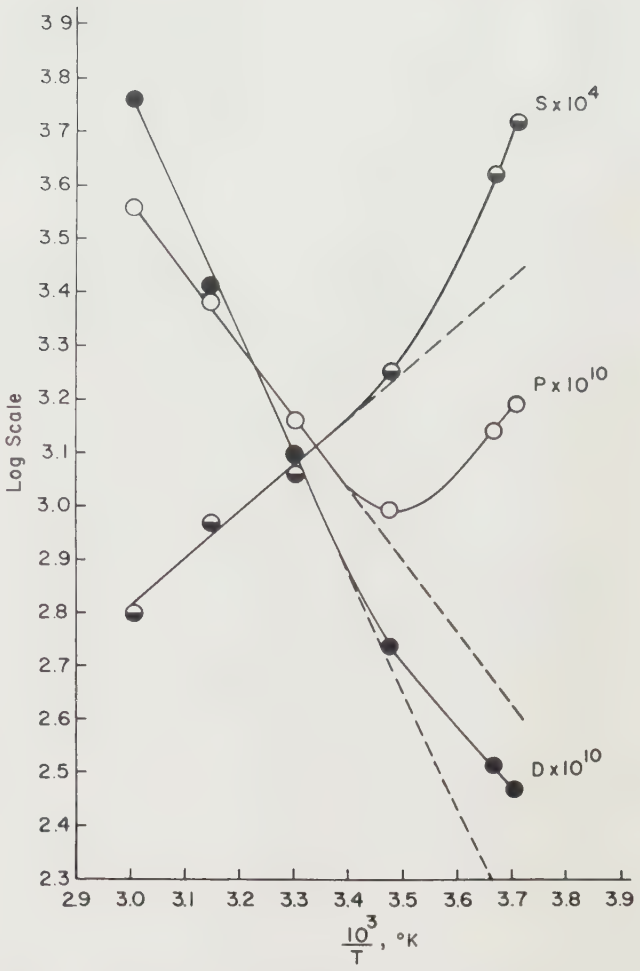

Fig. 9. Variation of P, D, and S with temperature. Methyl bromide (600 mm. Hg) and polyethylene (0.922 g./cc.)

first approximation, the magnitude of E_D and E_P will increase as the size, shape, and polarity of the penetrant increase and as the polarity (or cohesive energy density) of the polymer increases. These trends of E_D and E_P are related to the energy requirements for hole formation in the polymer matrix. Some of the more specific effects and interpretations have been discussed in preceding sections.

The temperature dependences of the diffusion and permeability constants usually are such that P and D decrease uniformly with decrease in temperature. However, in some temperature–pressure regions the permeability constant sharply increases as the temperature decreases (*9*).

Figure 8 shows the temperature dependence of P for methyl bromide in polyethylene for three different pressures. The isobars corresponding to higher pressures deviate widely from the normal linear relationship, the value of P first decreasing and then increasing as the temperature is progressively lowered. At sufficiently low pressure, normal linear plots are obtained over the temperature range investigated.

Independent determinations of the temperature dependences of D and S for methyl bromide in polyethylene at a pressure of 600 mm. Hg show that at the lower temperatures both D and S depart from the normal linear relationships, Fig. 9.

It has been shown experimentally (*9*) that the main contribution to the heat of solution of methyl bromide in polyethylene comes from the heat of condensation of

Table VI. Effect of Sorbed Vapor on Energy of Activation for Diffusion in Polyethylene (Density 0.922 g./cc.)

$p/p°$	E_D, kcal./mole	
	Methyl bromide	*Isobutylene*
0.0	13.9	17.8
0.5	11.9	15.1
1.0	6.7	10.5

the vapor, so that the solubilities at constant relative vapor pressure are virtually independent of temperature. Therefore, lowering the temperature at constant pressure leads to the same sequence of events as increasing the pressure at constant temperature; in both cases the activity of the vapor increases. As shown previously, the concentration of sorbed vapor in a polymer increases rapidly with increase in vapor activity; in like manner, the diffusion of the vapor is exponentially dependent on the sorbed vapor concentration or activity. In the methyl bromide–polyethylene system the permeability isobar passes through a minimum at a vapor activity of about 0.5. The increase of D (and P) at the higher activities results from the plasticizing effect of the sorbed vapor. Furthermore, the activation energy for diffusion is diminished, Table VI, since the motion of polymer segments in the presence of plasticizer is less hindered than in its absence. The larger values for E_D of isobutylene reflect the larger size of the penetrating molecule.

The above phenomena should be observed in some temperature–pressure range in any system where the condensed vapor plasticizes the polymer sufficiently to make the diffusion process concentration-dependent. Indeed, similar behavior has been demonstrated in the systems: isobutylene–polyethylene (*9*), water vapor–nylon (*30*), and water vapor–polyvinyl alcohol (*30*).

SELECTIVE PERMEATION THROUGH COMPOSITE FILMS

In the preceding sections, it has been shown that the sorption, diffusion, and permeation of organic vapors in polymers often is strongly dependent on vapor activity and the concentration of sorbed penetrant. One of the many consequences of these phenomena is that the permeation of a vapor through a composite film barrier or films in series is thereby dependent on the sequence in which the films are assembled with respect to direction of the vapor flux.

For the ideal case, when there is no pressure-dependent permeation occurring in the system, the overall permeability constant, P, for an assembly of n films is given by the harmonic average of properly weighted permeability constants, P_i, of the individual layers (x_i/L being the respective weight).

$$\frac{1}{P} = \sum_{1}^{n} \left(\frac{x_i}{L}\right)\left(\frac{1}{P_i}\right) \qquad (16)$$

This result means that ideally the rate of permeation through a composite membrane is independent of the order in which the layers are assembled.

When any P_i is pressure-dependent equation (16) is invalid and the overall permeability is affected by the order in which the layers are assembled. If the concentration or pressure dependence of the vapor permeability is known for each of the individual films, then it is possible to predict the overall permeation rate for any combination or permutation of the individual films by a method of graphical analysis described in detail elsewhere (*31*). By this method it has been shown that for a membrane composed of two films, the permeability constant of one increasing rapidly with pressure while the permeability constant of the second only increases slightly (or not at all), the composite film will be more permeable if the first film is exposed to the high pressure than if the gradient of pressure is reversed. This conclusion has been verified (*31*) by using a membrane composed of nylon and ethyl cellulose films with water vapor as the penetrant. Experimental determinations show that there is reasonable agreement between the calculated and the observed rates of permeation, Table VII. The same general behavior would certainly be expected if the even more pressure-dependent organic vapor–polymer permeation systems were used.

Table VII. Transmission of Water Vapor at 25°C. Through Two Films in Series

Film	*Pressure, mm. Hg*	*Transmission, cc. STP/cm.²/sec.*	
		Experimental	*Calculated*
Nylon 6	23.0	89.2×10^{-6}	80.5×10^{-6}
↓	21.8	70.4	61.0
Ethyl cellulose	19.9	38.4	35.0
	19.3	30.9	29.0
	14.8	10.4	9.1
	4.5	1.7	1.7
Ethyl cellulose	22.7	26.2 ± 2.0	35.0
Nylon 6 ↓	20.5	18.1 ± 1.2	21.5
	17.4	12.6	11.8
	14.9	7.9	8.0
	10.2	4.0	4.0
	4.5	1.1	1.7

LITERATURE CITED

1. Rogers, C. E., Meyer, J. A., Stannett,V., and Szwarc, M., *Tappi* 39, 737, 741 (1956); 40, 142 (1957).
2. Pinsky, J., *Modern Plastics* 34, 145 (1957).
3. Martinovich, R. J. and Boeke, P. J., *Modern Packaging* 31, (1957).
4. Waack, R., Alex, N. H., Frisch, H. L., Stannett, V., and Szwarc, M., *Ind. Eng. Chem.* 47, 2524 (1955).
5. Barrer, R. M. and Skirrow, G., *J. Polymer Sci.* 3, 564 (1948).
6. Aitken, A. and Barrer, R. M., *Trans. Faraday Soc.* 51,1 16 (1955).
7. Sobolev, I., Meyer, J. A., Stannett, V., and Szwarc, M., *J. Polymer Sci.* 17, 417 (1955).
8. Bent, H. A., *J. Polymer Sci.* 24, 387 (1957).
9. Sobolev, I., Meyer, J. A., Stannett, V., and Szwarc, M., *Ind. Eng. Chem.* 49, 441 (1957).
10. Rogers, C. E., Myers, A. W., Stannett, V., and Szwarc, M., in "Plastics Progress" 45–58, London, Iliffe, 1958.
11. Myers, A. W., Rogers, C. E., Stannett, V., and Szwarc, M., *Tappi* 41, 716 (1958).
12. Rogers, C. E., Stannett, V., and Szwarc, M., *J. Phys. Chem.* 63, 1406 (1959).
13. Doty, P. M., Brownstein, M., and Schleuer, W., *J. Phys. and Colloid Chem.* 53, 213 (1949).
14. Richards, R. B., *Trans. Faraday Soc.* 42, 10 (1946).
15. Muthana, M. S. and Mark, H., *J. Polymer Sci.* 4, 527 (1949).
16. Barrer, R. M., Barrie, J. A., and Slater, J., *J. Polymer Sci.* 177 (1958).
17. Matsuoka, S., private communication.
18. Barrer, R. M., *J. Phys. Chem.* 61, 178 (1957).
19. Crank, J., "The Mathematics of Diffusion," London, Oxford Univ. Press, 1956.
20. Meares, P., *J. Polymer Sci.* 27, 391, 405 (1958).
21. Rogers, C. E., Stannett, V., and Szwarc, M., *J. Polymer Sci.* 40, 61 (1960).
22. Rogers, C. E., to be published.
23. Zimm, B. H. and Lundberg, J. L., *J. Phys. Chem.* 60, 425 (1956).
24. Park, G. S., *Trans. Faraday Soc.* 48, 11 (1952).
25. Cutler, J. A., Kaplan, E., McLaren, A. D., and Mark, H., *Tappi* 34, 404 (1951).
26. Prager, S. Long, R. A., *J. Am. Chem. Soc.* 73, 4072 (1951).
27. Kokes, R. J., and Long F. A., *J. Am. Chem. Soc.* 75, 6142 (1953).

28. Long, F. A. and Thompson, L. J., *J. Polymer Sci.* 14, 321 (1954).
29. Park, G. S., *Trans. Faraday Soc.* 46, 684 (1950); 47, 1007 (1951).
30. Myers, A. W., Meyer, J. A., Rogers, C. E., Stannett, V., and Szwarc, M., *Tappi* 44, 58 (1961).
31. Rogers, C. E., Stannett, V., and Szwarc, M., *Ind. Eng. Chem.* 49, 1933 (1957).
32. Newns, A. C., *J. Textile Inst.* 41, T269 (1950).
33. Flory, P. J., "Principles of Polymer Chemistry," Chap. XII. Ithaca, N. Y., Cornell Univ. Press, 1953.
34. Michaels, A. S. and Parker, R. B., Jr., *J. Polymer Sci.* 41, 53 (1959).
35. Thornton, E. R., Stannett, V., and Szwarc, M., *J. Polymer Sci.* 28, 465 (1958).

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Some Techniques for the Assessment of Coated Art Papers

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The quality of high grade coated art papers is such that it is not possible to make sudden outstanding improvements. Small improvements are often not confirmed by commercial printing trials because of variability in the printing. If a program of research into coated art paper is to succeed, small improvements must be detected in order that they can be recognized. The techniques of surface measurement of the sheet and of controlled printing, described in this report, have enabled such small improvements to be perceived. These techniques involve the use of conventional smoothness testers, and gloss meters, and also the use of a high sensitivity surface profile instrument, the I.G.T. printability tester and an instrumented Vandercook proofing press. To use the proofing press properly a scheme of print evaluation with a sufficiently high sensitivity had to be established. This was done by the use of a "paired comparison" technique. It was found that the surface profile test correlated best with print quality, and this test is now used for production control work. Very small improvements could now be recognized, and the results are aptly illustrated by the difference in printability of the art paper before and after the full investigation. This work involved assessment of over 50 changes to the materials and processes concerned, each of which was by itself undetectable in commercial printing trials.

By COATED art papers is meant those papers made from a furnish free from groundwood, which are coated off the paper machine—in American parlance, conversion-coated—by brush, reverse roll, air-knife, spreadshaft, contra-coaters, or other similar equipment. In this article emphasis is placed on those top-grade papers which are used for mutlicolor letterpress printing.

With the possible exception of cast-coated papers, such papers present the letterpress printer with the best surface he can find for his art. When the surface is accompanied by the necessary cushioning effect of the support, he can produce his finest quality prints. Such papers comprise only about 10% of the total production of coated paper. As they are already in the top quality, any improvements that can be made must of necessity be small ones.

In my company, a program of research into such papers has been carried out over the past five years. If one compares a halftone print on the paper before the research started, with one on the present production, the change is quite extraordinary, particularly if, as in Fig. 1, a very light impression is used.

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However, the change shown here is the result of combining very many small changes to the paper. When this work was started, and the first small change was made, the paper was sent to several printers for evaluation. The results obtained were quite contradictory. Some of the printers found the trial paper a great improvement, some found it worse than the original, and others could not see any difference. This type of result was obtained every time such small changes were made and it was soon realized that if the assessment of trials was to be based on the commercial printing runs, no definite judgment could be obtained.

It was, therefore, decided to set up a technique for the assessment of print quality, which should be capable of picking up very small differences.

PRINTABILITY TESTING

The apparatus available consisted of the normal laboratory instruments, an I.G.T. printability tester (*1*), a surface-profile tester (the Talysurf described in Appendix I), and a Vandercook proofing press.

An experiment was therefore set up to try to find a way of operating the Vandercook press so that small differences were readily detectable, and to try to correlate the results found with those of I.G.T. testing and physical measurement. To do this, 11 British coated art papers were selected, which sold at the same price and which were reckoned by the trade to be only marginally different in quality. The quality of a print will

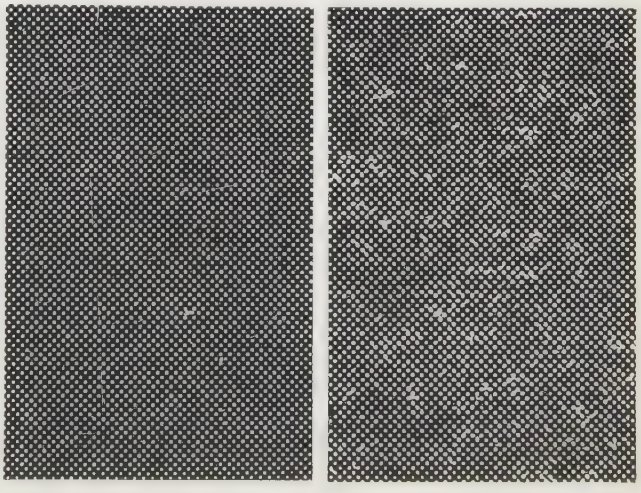


Fig. 1. Halftone print on art paper before (left) and after development work

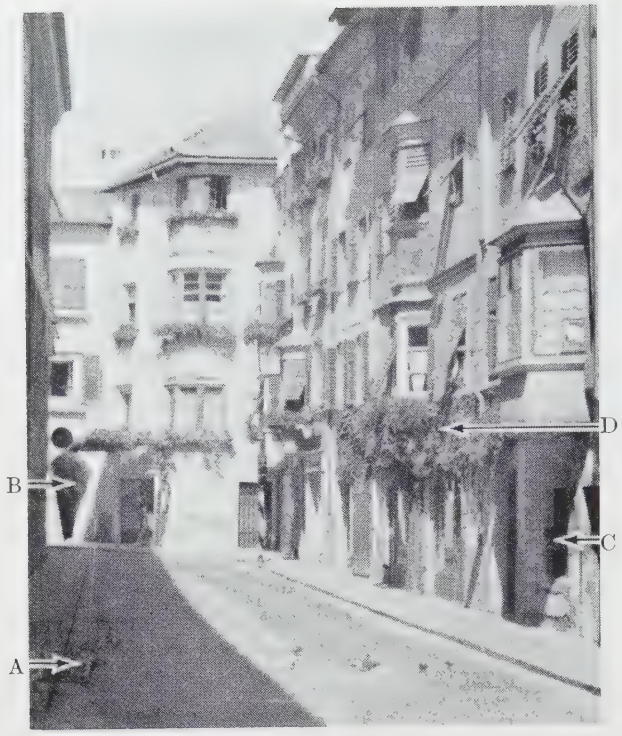


Fig. 2. The Vandercook test print

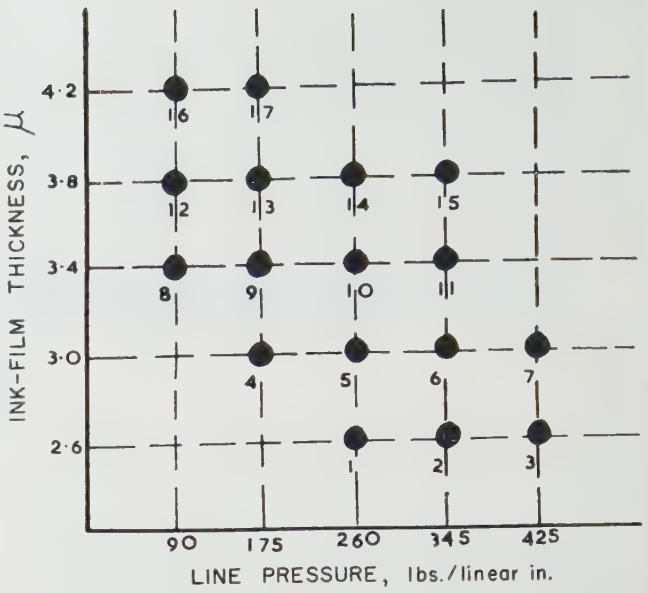
depend upon the paper, the ink, the plate, the impression, and the ink film thickness.

The Ink. After much discussion with the inkmakers, and with printers, it was finally decided to ask an inkmaker to recommend a suitable ink. One was found, and used straight from the tin, without further doctoring.

The Plate. Coated art papers are produced for fine screen halftone work. With the help of PATRA. (*2*) a subject containing a wide range of tone values was selected, with a screen ruling of 133 lines per inch—the ruling most commonly used in the British Isles for this type of work. Figure 2 shows a print from the plate finally selected.

Impression and Ink-Film Thickness. Most printers try to adjust these to obtain the best combination for the paper being used. In print-testing laboratories, however, impression and ink-film thickness are usually standardized so that all prints for comparison are made under the same conditions. Usually conditions of low impression and low ink-film thickness are selected, as in Fig. 1, so that defects in the paper may be easily shown up. This is obviously most convenient from a testing aspect, but it is quite possible that differences between papers, which may be apparent at these extreme conditions, are of negligible importance when the papers are printed under the conditions pertaining to normal commercial practice. The printer's skill, too, will enable him to make ready his machine to suit the particular paper being printed. It is highly likely that papers printed under one set of conditions will be ranked differently from those printed under another, and differently again if each is printed under the optimum conditions for that paper.

It was decided, therefore, to plan an experiment so that the papers could be printed at various standard conditions, and also so that the optimum printing condition for each paper might be found. To do this,



Each circle represents a combination of impression and ink-film thickness used in experiment.

Fig. 3. Printing conditions in factorial printing experiment

a plan of printing at various impressions and at various ink-film thicknesses was set up, as shown in Fig. 3.

Printing Procedure. Previous work had provided means of determining the amounts of ink to be added after each run to maintain the ink-film thickness reasonably constant. The printing practice adopted for each run was:

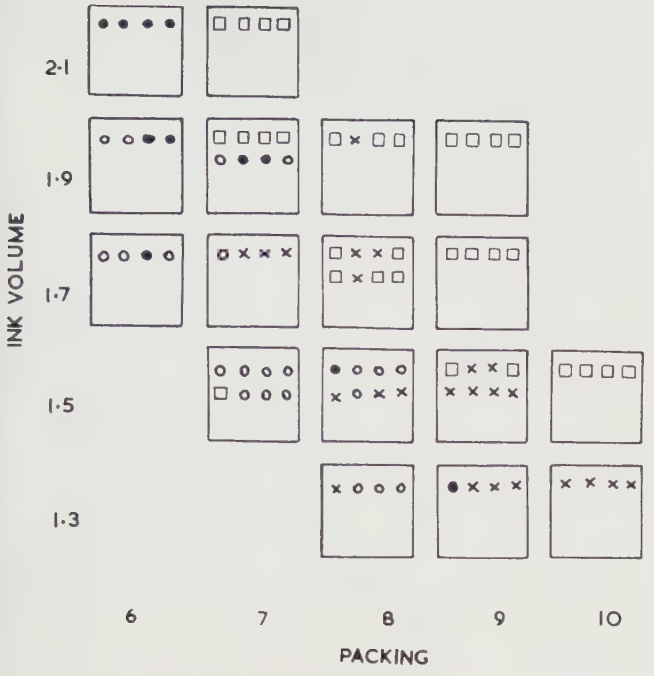
(a) Pack the cylinder and run this with paper across the plate several times to bed the packing down.
(b) Apply ink to the distribution system and let it run for 15 min.
(c) Run six prints on standard paper to condition the plate.
(d) Run the prints on the paper being tested, interleaving the test sheets with standard paper, so that there could be no interaction from one sheet to the next.
(e) Make ink additions after every second print to keep the ink supply constant.

Experimental Design. Nine printing conditions were investigated in the first experiment—nos. 4, 5, 6, 9, 10, 11, 13, 14, and 15 of Fig. 3. Each make of paper was printed under these nine conditions, and two duplicate prints at two of the conditions selected randomly were also done. Thus, paper 1 might be printed at conditions 4, 5, 6, 9, 10, 11, 13, 14, 15 with repeats at conditions 5 and 9. Paper 2's repeats might be at conditions 4 and 11, etc.

The 11 makes of paper were printed at the above conditions but the order in which they were printed was randomly varied from condition-to-condition.

Each print was coded 1 to 11 to identify the printing condition. However, each make of paper was given a different code for the printing conditions in order that the print assessors might not identify any one code number with any one printing condition.

The initial choice of printing conditions covered the block of medium ink-film thicknesses and medium printing pressures. When prints from this experiment were analyzed it was found that for some papers, opti-



○ Grain. □ Filling-in. ● Grain and filling-in. × Satisfactory.

Fig. 4. Initial selection of prints for ranking judgment of four observers

mum printing conditions had not been reached, so a second experiment was carried out on the same lines, but at printing conditions 1, 2, 3, 7, 8, 12, 16, 17, together with 6, 10, and 13 of the original range for comparison purposes with the first experiment.

ASSESSMENT OF THE PRINTS

Print assessment carried out by four experienced observers. These four had been selected as a result of previous work *(3, 4)*.

It was found, particularly in these earlier investigations, that "lay" observer—those with no practical experience of print quality—rank as best those prints which are overprinted. They are very conscious of grainness and lack of contrast, but have no concept of filling-in of the halftone screen or of the presence or absence of tonal gradation. It is interesting to note what the experienced observers look for. They used particularly the areas marked *A*, *B*, *C*, and *D* in the print (Fig. 2). One went so far as to seek the pigeon in area *A*.

Each make of paper, therefore, provided 22 printed sheets, and for each make, the 22 prints were examined independently by each observer and classified as follows:

(a) Prints unsatisfactorily grainy.
(b) Prints unsatisfactorily filled in.

Figure 4 shows the typical result for one paper, with the four observers.

For the next ranking, every print (out of the 22 on each paper) which had been considered as satisfactory by at least one observer was included.

Thus for each of the 11 makes of paper, a set of prints was assembled. This set was then ranked independently by three observers, using a paired-comparison technique. This technique allows a score to be given to a print—the number of other prints it is better than—and is described in Appendix II. The scores obtained by each observer were added together, giving each

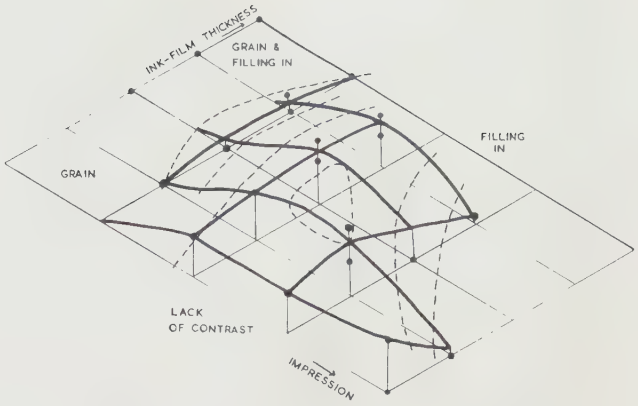


Fig. 5. Print quality response surface

sheet a numerical value. As each sheet was already defined by an ink-film thickness and impression figure, for each make of paper a response surface was built up. Figure 5 shows such a reponse surface for one of the makes of paper.

By using the response surface and plotting-in the contours, the optimum printing condition for each make of paper could thus be determined. Table I gives the actual condition for optimum printing for each paper.

Finally, to obtain the ranking between the makes of paper at optimum printing conditions for each, the print for each make, selected as described above, was ranked against the others by the four observers. The paired-comparison technique was again used and the ranked results are given in Table II. It will be noticed that the results are given for each observer separately, and that the coefficient of concordance *(5)* between observers is 0.87; 1 being perfect agreement.

The next thing was to establish the agreement of the "optimum" rankings with the rankings obtained by printing at standard conditions. Conditions 4, 5, and 6, and 7 of Fig. 3 were taken. Three observers were used, and their rankings are given in Table III. Again it can be seen that agreement between observers is extremely high.

Next, because of the good agreement between observers, the mean ranks of the papers for printing conditions 4, 5, 6, and 7 have been taken and compared with the ranking of the papers at the optimum printing conditions. This has been done in Table IV. This time Spearmans rank correlation coefficient has been used to assess agreement between rankings. It can be seen that there is good agreement between the ranking of prints at printing conditions 5, 6, and 7, and that each of these correlates reasonably well with the ranking of prints at optimum printing conditions.

Table I. Optimum Printing Conditions

Paper	*Position no.*	*Cylinder packing*	*Ink volume, cc.*
1	6	9	1.5
2	10	8	1.7
3	3	10	1.3
4	3	10	1.3
5	6	9	1.5
6	6	9	1.5
7	3	10	1.3
8	3	10	1.3
9	6	9	1.5
10	6	9	1.5
11	3	10	1.3

Table II. Ranking of Prints at Optimum Conditions

Coefficient of concordance (W) = 0.87

Paper	Observer 1	Observer 2	Observer 3	Observer 4	Total	Overall rank
1	3	2	3	4	12	3
2	5	4	6	3	18	4
3	8	10	10	7	35	8.5
4	7	8	7	5.5	27.5	7
5	1	1	3	1	6	1
6	2	3	3	2	10	2
7	10	8	8	10	36	10
8	6	5	5	5.5	21.5	6
9	9	8	9	9	35	8.5
10	4	6	1	8	19	5
11	11	11	11	11	33	11

Table III. Ranking Prints Printed at the Same Condition

Printing condition no. →	Coefficient of Concordance (W)											
Observer →	0.94			0.97			0.99			0.89		
	4			5			6			7		
Paper	1	2	3	1	2	3	1	2	3	1	2	3
1	6	6	6	7	6	5	5	3.5	5	6	7	6
2	7	8	7	3	3	3	4	2	3	3	3	7.5
3	9	9	10	10	10	10	10	8	9	9	9	9
4	5	5	4	5	7	6	7	5.5	6	8	8	7.5
5	1	2	2	1	2	2	1	3.5	2	1	2	2
6	2	1	1	2	1	1	2	1	1	2	1	4
7	10	11	9	8	9	9	8	10	9	10	10	10
8	3	3	5	4	4	4	3	5.5	4	4	4	2
9	8	7	8	9	8	8	9	9	9	7	6	5
10	4	4	3	6	5	7	6	7	7	5	5	2
11	11	10	11	11	11	11	11	11	11	11	11	11

Table IV. Rankings of Prints at Individual Positions

Mean rankings of three observers

Paper	Rank correlation with optimum condition ranking (R) 0.76 (4)	0.87 (5)	0.86 (6)	0.84 (7)	Mean ranking for 4 conditions	Optimum condition ranking
	Printing condition no.					
1	6	6	6.5	6.3	6.2	3
2	7.3	3	3	4.5	4.4	4
3	9.3	10	9	9	9.3	8.5
4	4.7	6	6.2	7.8	6.2	7
5	1.7	1.7	2.2	1.7	1.8	1
6	1.3	1.3	1.3	2.3	1.5	2
7	10	8.7	9	10	9.4	10
8	3.7	4	4.2	3.3	3.8	6
9	7.7	8.3	9	6	7.8	8.5
10	3.7	6	6.7	4	5.1	5
11	10.7	11	11	11	10.9	11

It must be remembered in assessing these results that these papers are all sold at the same price for the same purpose and that one would not expect large printability differences between them. In view of this, the agreement between ranks after printing at standard conditions and ranks after printing at optimum conditions is very good indeed and fully justifies the use of standard printing conditions for print assessment. While standard conditions are used in very many laboratories, it is believed that this is the first time that such agreement has been proved.

One interesting result is obtained when the ranking at optimum printing conditions is compared with the optimum condition found. This is given in Table V. It can be seen that, in general, the poorer papers have as their optimum printing condition no. 3; i.e., high pressure must be applied to prevent grainy prints, and ink-film thickness must be reduced to prevent filling-in of the halftone screen.

This work showed that it was possible, by following a standard printing technique, to rank papers for quality

Table V. Optimum Printing Conditions of Papers in Optimum Ranking

Paper	Optimum printing condition	Optimum condition rank
5	6	1
6	6	2
1	6	3
2	10	4
10	6	5
8	3	6
4	3	7
3	3	8.5
9	6	8.5
7	3	10
11	3	11

of print, even though only very small differences existed between them. However, printing in such a way takes up considerable time. This is still true, even though the proofing press has now been equipped with a pressure-loading device which allows constant pressure printing to be carried out, and a timing device which allows constant printing speed.

Physical Testing

For quality control purposes, therefore, it is essential that tests be formed which will give an indication of the printing quality of the paper within a few minutes of the paper being made. A physical testing program was therfore carried out on the papers mentioned above, the tests are given in Table VI.

The full results of all these tests are not given here, as they fill several pages of tables. However, correlation coefficients were obtained between the test results and the print quality rankings. Two print-quality rankings have been used: the "optimum print" ranking and

Table VI. Physical Testing

	No. of sheets taken per ream	No. of tests per sheet	No. of tests per ream
Moisture content as received, %	2	2	4
Substance, g.s.m.	5	1	5
Thickness, mils	5	5	25
Printing opacity, %	5	1	5
Contrast ratio, % of $MgCO_3$	5	1	5
Brightness, Eel filter %			
Top side	5	1	5
Wire side	5	1	5
Gloss, Gardner 85° head %			
Top side	5	1	5
Wire side	5	1	5
Surface profile, Talysurf micro-inches			
Top side	5	4	20
Wire side	5	4	20
Bendsten roughness, cc./min.			
Top side	5	1	5
Wire side	5	1	5
Bekk smoothness, sec.			
Top side	5	1	5
Wire side	5	1	5
I.G.T. pick, cm./sec.			
Top side	5	2	10
Wire side	5	2	10
I.G.T. print density, ratio			
Top side	5	1	5
Wire side	5	1	5
Dennison wax, no.			
Top side	5	1	5
Wire side	5	1	5
K & N ink, % brightness			
Top side	5	1	5
Wire side	5	1	5
Furnish,	2	1	2
Coating weight, g.s.m.	2	1	2

Table VII. Correlation of Optimum Condition Ranking and Overall Print Ranking with Ranking by Other Methods

R is Spearman's rank correlation coefficient

Method	Optimum cond. ranking (R)	Overall print ranking (R)
Overall print ranking	+0.91[a]	...
Visual assessment of prints		
Condition 4	+0.85[a]	+0.92[a]
Condition 5	+0.90[a]	+0.97[a]
Condition 6	+0.92[a]	+0.95[a]
Condition 7	+0.84[a]	+0.95[a]
Substance (ranked low, high)	+0.33	+0.27
Thickness (ranked low, high)	+0.21	+0.26
Wire side gloss (ranked low, high)	−0.34	−0.22
Wire side brightness (ranked low, high)	+0.05	+0.12
Contrast ratio (ranked low, high)	+0.32	+0.14
Printing opacity (ranked low, high)	−0.27	−0.45
Bulk (ranked low, high)	+0.06	+0.15
Ash of base paper (ranked low, high)	−0.30	−0.34
Wire side K&N (reflect. paper-ref. stain) (ranked low, high)	−0.08	−0.01
Wire side K&N (reflect. stain/ref. paper) (ranked low, high)	+0.07	0
Ash of coated paper (ranked low, high)	+0.57[b]	+0.71[c]
Moisture content of ream (ranked low, high)	−0.43	−0.60[b]
Wire side smoothness Bekk (ranked low, high)	−0.48	−0.64[b]
Wire side smoothness Bendsten (ranked low, high)	+0.79[c]	+0.80[c]
Base paper, % (ranked low, high)	−0.65[b]	−0.74[c]
Wire side smoothness Talysurf MD (ranked low, high)	+0.81[c]	+0.91[a]
Wire side smoothness Talysurf XD (ranked low, high)	+0.86[a]	+0.87[a]
Wire side smoothness Talysurf mean (ranked low, high)	+0.83[a]	+0.87[a]
Wire side print contrast (ranked low high)	−0.71[c]	−0.82[c]
Visual assessment of I.G.T. prints		
Set 1	+0.71[c]	+0.86[a]
Set 2	+0.57[b]	+0.62[b]
Set 3	+0.74[c]	+0.85[a]
Set 4	+0.85[a]	+0.89[a]

[a] R is significant at better than 0.1% levels (0.000 $R \geq 0.825$).
[b] R is significant between 5 and 1% levels ($0.523 \leq R \leq 0.825$).
[c] R is significant between 1 and 0.1% levels ($0.683 \leq R \leq 0.825$).

the overall ranking, the latter being the mean of the rankings obtained at printing conditions 4, 5, 6, and 7.

The correlation coefficient used is Spearmans rank correlation coefficient and the results are given in Table VII.

As can be seen from this table, there are significant correlations between the print quality rankings and I.G.T. prints both visually ranked and by print density, Bendsten and Bekk smoothness, Talysurf surface profile, and percentage base paper. There was no significant correlation with substance, thickness, bulk, gloss, contrast ratio, brightness, or K & N absorption.

A closer examination of these results, however, shows that not all these tests are suitable for assessment purposes. Quality control tests should be simple to carry out and quick to use. The time taken for any test will depend on the number of measurements necessary to give adequate precision to the mean value and on the time for each individual measurement. These factors are taken into account in the following assessment.

First, however, it must be stated that the correlation was with halftone print quality. Many of the properties that did not correlate with print quality would correlate with other aspects of printing. Gloss may be more important for large solids, and K & N absorbency certainly has a bearing on ink drying. Bulk, too, may be important from compressibility considerations.

Of those properties which did correlate with the print quality assessment the I.G.T. is another printing test, on a smaller scale, which one would expect to correlate, although in the I.G.T. test a solid, not a halftone, is printed. This method could well be used for quality control, although the test is by no means simple to carry out, and requires an experienced observer to judge results. The I.G.T. print density does away with the need for trained observers, but unfortunately is not practicable because of the need to dry the print and the time taken to do so.

The most important correlations were with roughness measurements. The correlation with Bekk smoothness is the lowest of these, and each individual measurement requires about 2 min. testing. This, together with the difficulties inherent in using the instrument, makes it unsuitable for control testing.

Both the Talysurf and the Bendsten would appear

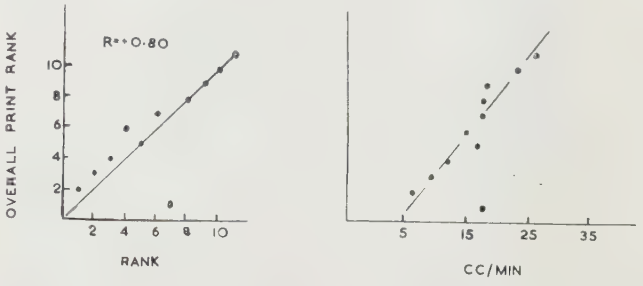


Fig. 6. Bendtsen roughness

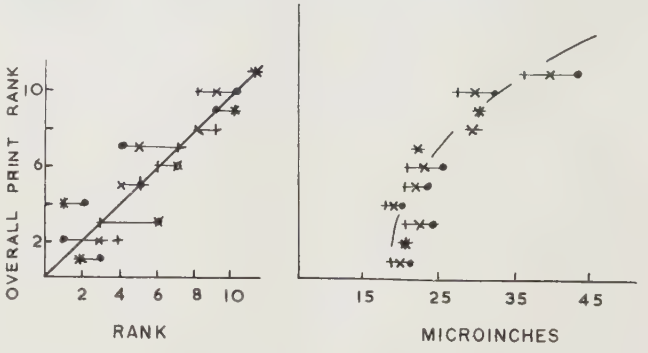


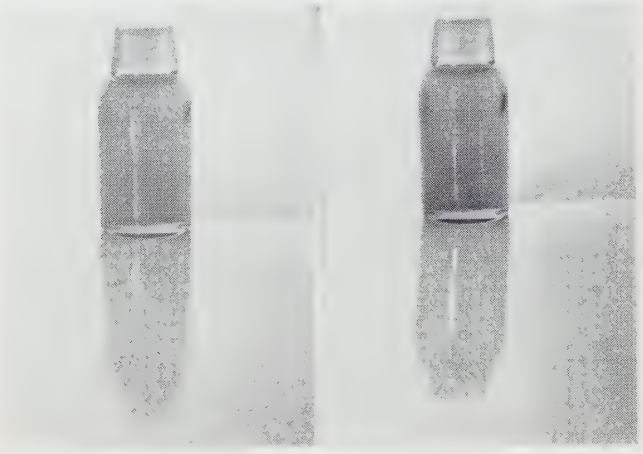
Fig. 7. Talysurf roughness (center line average)

to correlate well with the print quality rankings. Figures 6 and 7 demonstrate this. However, the average coefficient of variation of the Bendsten results is 25% for the top side and 64% for wire side. For the Talysurf it is 12% for the top side and 14% for the wire side. The average Bendsten difference between the papers is 2 cc. per min. and to show that two papers separated by this difference were significantly different 50 Bendsten measurements would be necessary on the top side and 320 on the wire side of each paper. For the Talysurf 10 and 12, respectively, would suffice. This definitely indicates the unsuitability of the Bendsten for quality control because of the time needed to do enough tests.

With the Talysurf, however, it can be seen from Fig. 7 that there is considerable difference in the results between the worst four papers and the remainder. This is in complete agreement with the results of the print quality assessment. It does highlight the in-

adequacies of ranking techniques, which record the order and not the degree of quality. The number of Talysurf measurements that would have been necessary to show that any one of these papers was significantly poorer than the poorest of the top six is only six—2 min. testing time per paper.

It would seem, therefore, that the Talysurf can be used as a quality control instrument. The instrument itself is soundly made, robust, and relatively foolproof in use. As a result of this work the Talysurf has been adopted as the routine instrument for quality control of art paper. Besides giving a "center line average" figure for control purposes, the Talysurf can also give a magnified profile picture. Some interesting pictures are shown in Figs. 8 and 9. Two papers were chosen: one brushed to give high gloss, and one cast-coated



Brushed Cast-coated

Fig. 8. Reflections in a brushed high gloss art and in a cast-coated paper

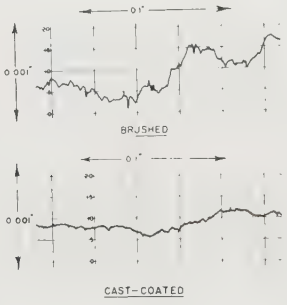


Fig. 9. Talysurf surface profile traces on brushed and cast-coated paper

paper. The difference in surface is shown up in Fig. 8 by photographing the reflection in the paper, of a bottle, and the Talysurf traces reproduced in Fig. 9 show the surface profile.

On this evidence and much more later work it can be asserted that in 99 cases out of 100, halftone print quality can be assessed by Talysurf measurements; assuming that all other factors, such as ink absorbency, are satisfactory.

It is also worth while commenting on two interesting results which arose from this investigation. The first—a highly significant correlation between base paper percentage and print quality—confirmed by a negative correlation between ash per cent in the coated paper and print quality. This, again, has been repeated since. It does depend very much on base paper quality, particularly compressibility, but is nevertheless there: i.e., within limits, a lick of coating is better than a thick layer.

Finally, there was a just significant correlation between print quality and the moisture content found on opening the reams. The correlation is poor, but would indicate that about 9% moisture should be aimed for.

CONCLUSIONS

The work carried out showed that if 11 different makes of high-grade art were assessed, there was a high degree of correlation between Vandercook prints obtained at the optimum degrees of impression and ink-film thickness for each paper, and those obtained when conditions of impression and ink-film thickness were standardized to be the same for each paper. This correlation was found to hold at four different standard conditions. The work also showed that the sensitivity of the printing was sufficient to pick up differences between papers which were sold at the same price for the same job.

The results also showed a significant correlation between smoothness and halftone printability. This correlation was greatest when a surface profile instrument, the Talysurf, was used to measure smoothness.

This instrument proved to be the best suited for quality control measuremenet on coated papers because of the low testing time (2 min.) required to obtain a precise result, and it is now in general use for this purpose.

APPENDIX I

The Talysurf

This is the proprietary name of an instrument which uses a stylus method for the recording of surface finish. It is widely used in the metal industry, and conforms closely to British Standard 1134:1950 and American Standard B 46:1955. It is manufactured by Taylor, Taylor and Hobson, Ltd., Leicester, England.

The measuring length, over which the stylus is drawn, is about 0.2 in., and the stylus tip radius is between 0.00014 and 0.0002 in. The stylus load is between 0.2 and 0.4 g.

The instrument will draw a profile of the surface examined at magnification of 1000, 2000, 5000, 10,000, 20,000, and 50,000.

It can also be used to give a centre line average—the average distance of the irregularities from their own center line, integrated over a length of surface of 0.03 in. This is the method used to give a numerical result for quality control purposes. Four measuring ranges can be selected, 0 to 6, 0 to 20, 0 to 60, and 0 to 200 microinches.

APPENDIX II

Paired Comparisons

Given, for example, three papers, *A*, *B*, and *C*. Rank *A* against *B* and *C*, and *B* against *C*. If *A* is better than *B*, score *A 1* and *B* 0. If *A* is worse than *B*, score A 0 and *B 1*. Thus if *A* is better than *B* and *C*, and *B* is better than *C*, *A* scores *2*, *B* scores *1* and *C* scores 0.

LITERATURE CITED

1. "The Printability of Paper," Manual of the I.G.T. Printability Tester. Institute for the Graphic and Allied Industries, Amsterdam, Holland. Publication 12.
2. PATRA. Printing, Packaging and Allied Trades Reserch Association, Great Britain.
3. Poulter, S. R. C., *International Bull.* No. 80: 49–51 (June, 1958).
4. Abbott, P. H. J. and Hendry, I. F., "Methods of Evaluating Art Papers for Letter-press Printability." EUCEPA 3rd Symposiun, Paris, October, 1959, 321–345.
5. Kendall, M. G., "Rank Correlation Methods," London, Griffin, 1955.

RECEIVED April 10, 1961. Presented at the 12th Coating Conference of the Technical Association of the Pulp and Paper Industry, held in Buffalo, N. Y., May 8–10, 1961.

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Stabilization of Peroxide Bleach Liquors with Organic Chelating Agents

Bleaching of Groundwood Pulp

R. D. SPITZ

The prevention of heavy-metal-ion-catalyzed decomposition of H_2O_2 in alkaline peroxide solutions with organic chelating agents of the aminopolycarboxylate type was studied. Pentasodium diethylenetriaminepentaacetate (Na_5DTPA) appeared to be the most effective of the commercially available aminopolycarboxylate chelating agents in reducing such hydrogen peroxide decomposition. This stabilizing effect of Na_5DTPA was applied to the peroxide bleaching of groundwood pulp by pretreating the groundwood slurry with Na_5DTPA prior to thickening and bleaching. Pretreatment of the groundwood slurry in this manner resulted in an increase in the peroxide-bleached brightness of 2 to 3 G.E. units in conventional peroxide bleaching systems. In addition significant reductions in brightness reversion of the bleached pulp were noted. This technique promises to be a useful addition to peroxide bleaching technology.

ALKALINE hydrogen peroxide bleach liquors which are employed extensively in the pulp and paper industry for the bleaching of groundwood pulp, and to a lesser extent for the bleaching of chemical pulps, are subject to decomposition. For example, Martin (*1*) in studying the effect of variables on the bleaching of eastern spruce groundwood with hydrogen peroxide found that some 20 to 30% of the peroxide added to the pulp is "lost" through decomposition and, presumably, serves no useful purpose in terms of the desired bleaching reaction. Furthermore, Nicoll and Smith (*2*) investigated the stability of alkaline hydrogen peroxide solutions and reported that the loss of active oxygen from such systems was primarily a function of the presence of catalytically active heavy metallic ions (Cu^{2+}, Mn^{2+}, Fe^{3+}, etc.). Reported herein are the results of laboratory investigations concerning the relative effectiveness of a number of commercially available chelating agents of the aminopolycarboxylate type in the prevention of heavy-metal-ion-catalyzed peroxide decomposition, and some mill-trial results concerning the pretreatment of groundwood pulp with pentasodium diethylenetriaminepentaacetate (Na_5DTPA) in conjunction with peroxide bleaching.

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LABORATORY EVALUATIONS

In the laboratory, the relative effectiveness of a number of compounds of the aminopolycarboxylate family of chelating agents in preventing heavy-metal-ion-catalyzed peroxide decomposition in simple aqueous solutions of peroxide was determined. The solutions used were prepared from deionized water and contained 0.35% H_2O_2, 1.0% of 42° Bé. sodium silicate and 0.25 p.p.m. copper ion. The various chelating agents, when added, were used at concentrations of 0.08 millimoles per liter. In each case the solution to be tested was held at a constant temperature (180°F.) and samples were withdrawn periodically, acidified with sulfuric acid, and titrated with standard potassium permanganate to determine the amount of residual hydrogen peroxide. The percentage of residual hydrogen peroxide was plotted as a function of time, an early set of data being shown in Fig. 1. The addition of Na_5DTPA at 0.08 millimoles per liter to the hydrogen peroxide solutions described above extended appreciably the life of the bleach solution. Other chelating agents evaluated in this series of experiments included tetrasodium ethylenediaminetetraacetate (Na_4EDTA), trisodium hydroxyethylethylenediaminetriacetate (Na_3HEDTA), disodium hydroxyethyliminodiacetate (Na_2HEIDA), trisodium ni-

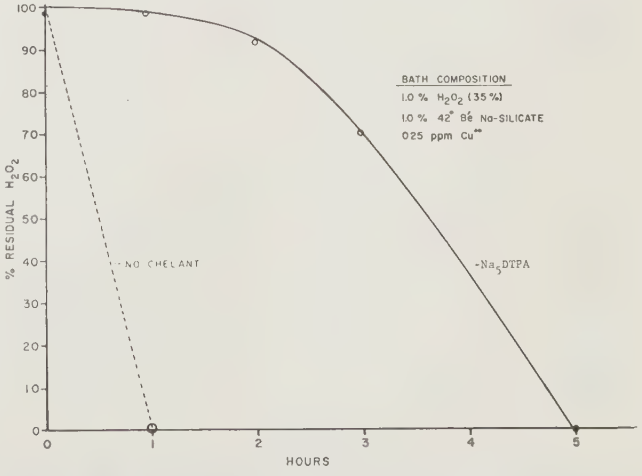

Fig. 1. Effect of chelating agents on stability of hydrogen peroxide solutions at a zero hardness level

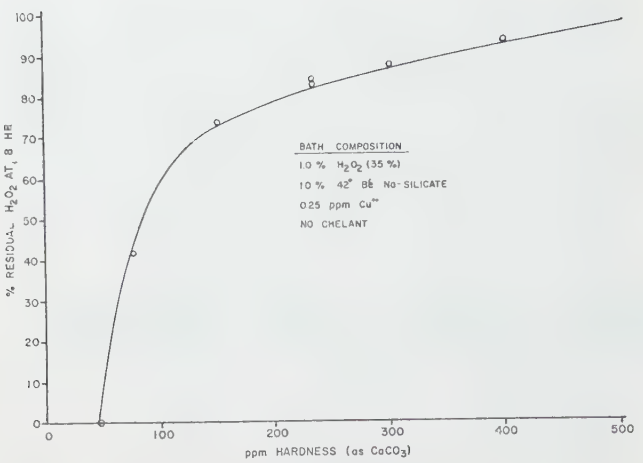

Fig. 2. Effect of hardness level on stability of hydrogen peroxide solutions

trilotriacetate (Na_3NTA), and triethanolamine (TEA). All these agents gave results which, if plotted in Fig. 1, would give a line nearly coincident with that shown for "no chelant."

Hence, Na_5DTPA appeared to be unique in its ability to prevent the decomposition of hydrogen peroxide catalyzed by a trace amount of copper ion. However, the applicability of the data was questioned since it would seem to indicate extremely poor stability of sodium silicate-containing peroxide bleach liquors in the presence of a trace of copper ion. Obviously, hydrogen peroxide solutions of such poor stability could not be used commercially for the bleaching of wood pulp.

Upon reviewing the literature on peroxide stability, numerous references were found concerning the profound effect of hardness ions upon peroxide stability. Apparently, sodium silicate, itself, is not an effective stabilizer in alkaline hydrogen peroxide bleach liquors. Rather, it would appear that calcium and magnesium silicates formed from the reaction of the sodium silicate with hardness ions tend to stabilize the hydrogen peroxide against heavy-metal-ion-catalyzed decomposition. The effect of hardness ions in the above system in the absence of chelating agent is shown in Fig. 2. The bleach solution composition described above was used and the graph shows the percentage of residual hydrogen peroxide remaining at a given time (8 hr.) as a function of the hardness level of the bleach solution. The stability of the hydrogen peroxide increases markedly with increasing hardness, especially in the 0 to 150 p.p.m. hardness range. Obviously, the absence of hardness ion in the earlier experiments was responsible for the lack of stability of the hydrogen peroxide solutions observed in the absence of chelating agent.

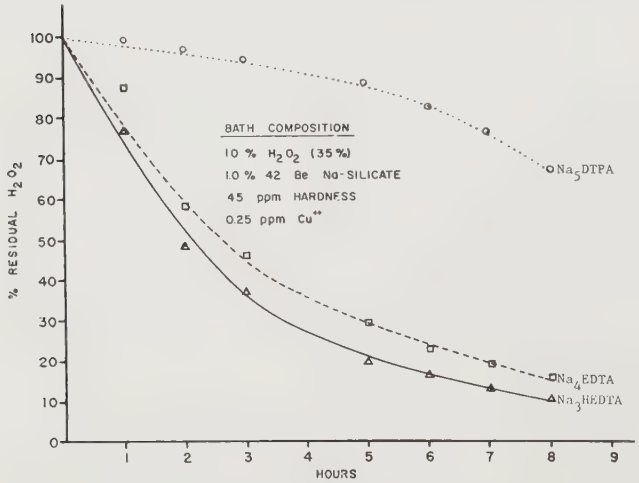

Fig. 3. Effect of chelating agents on stability of hydrogen peroxide solutions at a 45 p.p.m. hardness level

The aforementioned chelating agents were re-evaluated at two hardness levels—45 and 150 p.p.m.—by the procedure described above. Figure 3 shows the results obtained using Na_5DTPA, Na_4EDTA, and Na_3HEDTA as stabilizers at the 45 p.p.m. hardness level. Blank runs (no chelant) and runs using Na_2HEIDA, Na_3NTA, and TEA as stabilizers gave results very similar to those indicated for Na_3HEDTA. The data indicate that Na_5DTPA was by far the most effective of the chelating agents evaluated in the prevention of copper-ion-catalyzed peroxide decomposition while Na_4EDTA was of marginal utility and Na_3HEDTA, along with the other chelating agents evaluated, was of little or no utility. Figure 4 shows the data obtained at the 150 p.p.m. level. All the curves are displaced upward (greater stability) at the higher

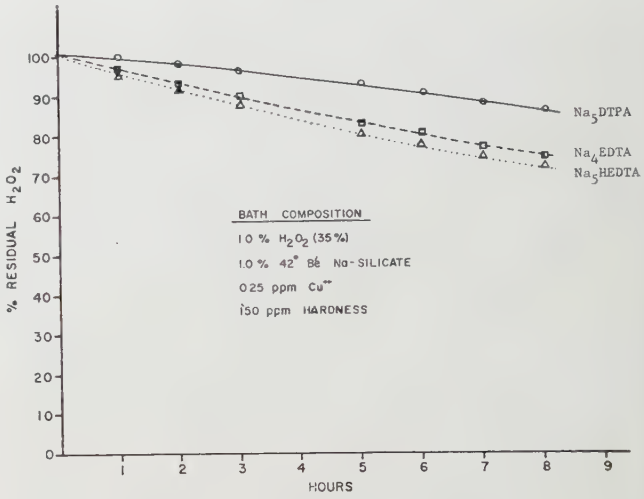

Fig. 4. Effect of chelating agents on stability of hydrogen peroxide solutions at a 150 p.p.m. hardness level

hardness level, but the relative position of the curves is preserved. Na_5DTPA still appeared to be the most effective with Na_4EDTA being of marginal utility and other agents evaluated, of little or no utility in the system studies.

At the 1959 TAPPI Convention Ian MacGugan reported the effect of various chemical pretreatments on the brightness development of groundwood pulp during peroxide bleaching. The pretreatment chemical evaluated were: calcium chloride, sodium tripolyphosphate, sodium bisulfite, peracetic acid, sulfuric acid, Na_4EDTA, and Na_5DTPA. They were added to the pulp slurry at 3% consistency and, after 10 min., the pulp was thickened to the desired bleaching consistency and bleached in the normal manner. Several important points are apparent from this work: (1) of the pretreatment chemicals evaluated, the organic chelating agents (Na_4EDTA and Na_5DTPA) were the most effective; (2) at equal weight loadings (10 lb. per ton of O.D. pulp) a 40% aqueous solution of Na_5DTPA was more effective than a 38% aqueous solution of Na_4EDTA; (3) in no case did any of the chemicals evaluated show any effect when added as an integral part of the bleach liquor rather than as a pretreatment.

Table I. Operating Conditions and Results of Mill Trial Using Versenex 80 in the Peroxide Bleaching of Groundwood

Conditions

	Bleach liquor composition[a]					Peroxide tower		
	Total peroxide as Na_2O_2, %	Sodium silicate 42° Bé., %	Total alkali as NaOH, %	STPP,[b] lb./ton	Versenex 80c, lb./ton	Temp., °F.	Retention time, min.	Consistency, %
2 Stages no Versenex	2.0	6.0	1.5	10	None	105	100	10–11
2 Stages with Versenex	2.0	4.0–4.5	1.8	None	6	120	90	10–11
1 Stage with Versenex	3.0	5.0	2.0	None	6	120	90	9–10

Results

		Brightness data, G.E.						
	Residual peroxide, %	Unbleached	Peroxide bleached	Peroxide increase	Hydrosulfite bleached	Hydrosulfite increase	Total increase	Bleached sulfite pulp added, %
2 Stages no Versenex	Approx. 20	59–61	71–72	11–12	76–77	5	16–17	30
2 Stages with Versenex	20–25	59–61	74–75	14–15	76–77	2	16–17	10–15
1 Stage with Versenex	20–25	59–61	76–77	16–17	...	...	16–17	0

[a] All percentages based on weight of O.D. pulp.
[b] Sodium tripolyphosphate
[c] Added at consistency regulator.

ORIGINAL MILL-TRIAL RESULTS

The present laboratory evaluations and the data reported by MacGugan strongly indicated that Na_5-DTPA was considerably more effective than other commercially available chelating agents in preventing the heavy-metallic-ion-catalyzed decomposition of peroxide. For the mill trials, therefore, Versenex* 80 (a 40% aqueous solution of Na_5DTPA) was used and reference hereafter to "the chelating agent solution" refers to this commercially available Na_5DTPA.

The first and most extensive mill trial using Na_5-DTPA for stabilizing alkaline hydrogen peroxide solutions in conjunction with the bleaching of groundwood pulp was conducted at a northeastern United States mill, bleaching groundwood pulp in a two stage (2% peroxide followed by 1% zinc hydrosulfite) bleach system. Figure 5 shows the physical setup of the two-state groundwood bleaching system studied. The pulp enters the bleach plant at 4 to 5% consistency and passes through the consistency regulator flowmeter where the consistency is adjusted to 3.0%. The pulp is then thickened to about 12% consistency at the screen thickeners, mixed with the hydrogen peroxide bleach liquor in the mixing chest, and allowed to pass through the first (peroxide) tower (approximately 10% consistency, 105 to 110° F., 90 min. retention time). Upon leaving the peroxide tower, the pulp is neutralized to pH 6 with SO_2, zinc hydrosulfite is added,and the pulp passes through the second (hydrosulfite) tower (140° F., 30 min). At the exit of the hydrosulfite tower the pulp is diluted to about 3% consistency and goes to storage and finally to the beaters where it is mixed with bleach and unbleached sulfite pulp for the machine furnish.

During the trial, the chelating agent solution was added to the pulp slurry near the entrance of the consistency regulator. It is important to note that the addition of the chelating agent in this manner reproduces the conditions MacGugan found most effective; i.e., *pretreatment* of the groundwood slurry prior to the addition of the alkaline peroxide bleach liquor. Chelating agents of the aminopolycarboxylate type chelate heavy metallic ions more readily under the slightly acidic conditions prevalent at the consistency regulator than under the alkaline conditions prevailing after the addition of the alkaline peroxide bleach liquor. Also, it is probably desirable to have the heavy metallic ion contaminants in the chelate form when the peroxide liquor is added.

Table I shows a condensed version of the results obtained during the trial at the subject mill. The use of the chelating agent solution at 6 lb. per ton of O.D. pulp, together with a higher pH and higher temperature, resulted in an additional 2 to 3 point brightness gain in the peroxide tower when operating on a two-stage system. The use of Na_5DTPA reduces the loss of peroxide as a result of decomposition and, therefore, a greater quantity of peroxide is available for the desired bleaching reaction. In order to make use of this greater quantity of available peroxide without increasing the retention time, it was found necessary to increase the pH and/or the temperature in the peroxide tower. The total brightness obtained from the two-stage system was not increased, since a drop in the increase in the hydrosulfite stage occurred. Attempts to get a greater brightness increase from the hydrosulfite stage by varying the conditions (pH, temperature) in the hydrosulfite tower were unsuccessful. Whenever a good brightness increase was obtained

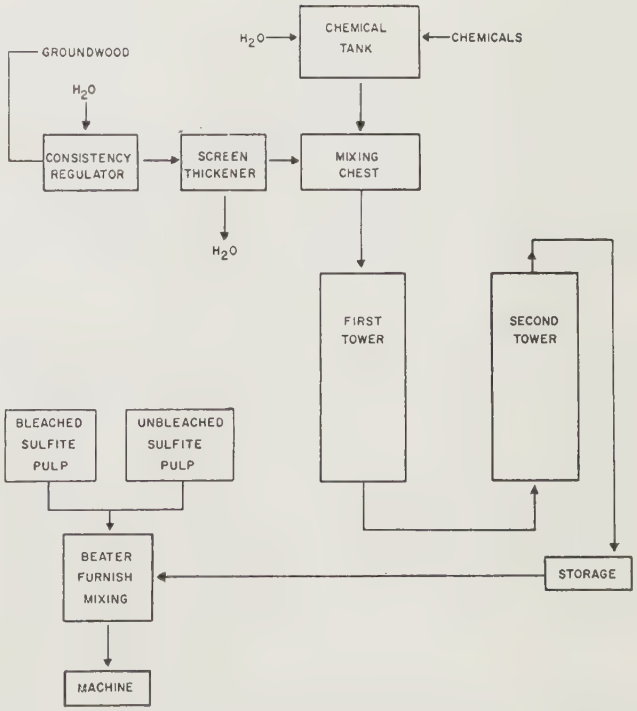

Fig. 5. Diagram of the two-stage bleach system studied

* Trademark of the Dow Chemical Co.

from the peroxide stage, no more than two additional points were obtained from the hydrosulfite stage.

The possibility of obtaining satisfactory brightness from a single (peroxide) stage by operating at a 3% sodium peroxide level while using the chelating agent solution a 6 lb. per ton of O.D. pulp was studied. With 3% sodium peroxide and a further increase in the sodium hydroxide addition, brightnesses obtained from the single stage were equivalent to those normally obtained from the two-stage system under similar operating conditions. The consistency in the peroxide tower was down somewhat at the 3% sodium peroxide level because of limitations in the chemical feed system. Had the normal 10 to 11% consistency been maintained, bleached brightnesses of 77 to 78 G.E. units would probably have been realized.

No appreciable savings in chemical costs resulted from operating with a single-stage system at a 3% sodium peroxide level, as opposed to a two-stage system at a 2% sodium peroxide level followed by 1% zinc hydrosulfite. Omission of the second stage resulted in some savings in steam costs, of course, due to the higher operating temperature normally employed in the second stage. The most significant savings brought about through the use of Na_5DTPA at this particular mill, however, resulted from the decreased quantity of bleached sulfite pulp required to maintain the specified off-the-machine brightness owing to the decrease in brightness reversion of the bleached pulp. Whenever Na_5DTPA was being used, the ratio or bleached to unbleached sulfite pulp added at the beaters was decreased considerably. Under normal operating conditions (two stages—no chelant) it was necessary to use about 30% bleached sulfite pulp in the paper machine furnish. When using Na_5DTPA in the two-stage system, the bleached sulfite pulp in the paper machine furnish could be reduced to approximately 15%, and when using Na_5DTPA and a single-stage bleaching system, brightness reversion was reduced even further and no bleached sulfite pulp was required. During the latter stages of the trial, significant amounts of unbleached groundwood (as high as 25%) were being added to the furnish.

RESULTS OF SUBSEQUENT MILL TRIALS

Following the above trial others were conducted at a number of groundwood mills located in the northeastern and midwestern United States. The bleaching systems encountered were, in general, single stage, medium density groundwood bleach ranges utilizing 1.5 to 3.0% sodium peroxide. The groundwood was pretreated in the manner described above with the chelating agent solution at levels of 5 to 10 lb. per ton of O.D. pulp. In these trials the use of Na_5DTPA increased the brightness of the bleached groundwood pulp by 2 to 3 units. Increases in off-the-machine brightness, in general, were greater than anticipated from the increased brightness of the bleached groundwood, due, presumably, to decreased brightness reversion of the bleached pulp.

LITERATURE CITED

1. Martin, M., *Tappi* 40, No. 2: 65–75 (1957).
2. Nicoll, W. D. and Smith, A. F., *Ind. Eng. Chem.* 47, No. 12: 2548–2554 (1955).

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Mill Operational Experiences Using the Stickle Pressure–Tension Moisture Control

WILLIAM F. JUDGE

The "heart" of any moisture control system is its sensing device, in this instance the tension roll. The theory of operation of the pressure-tension moisture control (Pres-Ten-Trol) is based on the fact that a change in moisture of the sheet will cause a corresponding change in the sheet tension. If a sheet carrying roll is so positioned that it is affected by this change in tension and moves correspondingly, then a moisture-sensing means has been arrived at.

The Bartgis Division of The New Haven Board & Carton Co. at Ilchester, Md., has two Stickle pressure-tension (Pres-Ten-Trol)* moisture controls installed on its two cylinder machines. These controls were purchased in 1956 and 1957. The two, six-cylinder machines, no. 2 and no. 1, trim 106 and 67 in., respectively. Each machine has 90 driers (mostly stack driers six-high) and uses turbine exhaust steam plus live makeup steam at the dry end, which is regulated by a pneumatically controlled, quick-opening steam valve. This valve is responsive to a signal from the Stickle control. The steam supplying the driers passes through two pressure-differential valves in supplying different drier stacks, and the condensate from the dry-end driers goes to two flash tanks where the flash steam is reused on the wet-end driers. The drainage system is of the microorifice glass-type.

The manufactured board varies in caliper from 16 to 40 mils. The grades of board vary from white patent coated to chipboard, and include bleached manila, single manila, clay-coated boards, and colored grades.

PRINCIPLE OF OPERATION OF THE PRES-TEN-TROL

The Stickle control is based on the principle that a change in moisture in the board will change the tension in the board. If a sheet-carrying roll is positioned so that this change in tension will cause a change in roll position, then a moisture sensing means has been arrived at. This sheet-carrying roll is called a tension roll. Basically, a movement caused by a change in moisture is changed to a pneumatic signal which regulates a steam valve.

The heart of the control, therefore, is its sensing device and the attached equipment which translates me-

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* Trademark of The Stickle Steam Specialties Co., Inc., Indianapolis, Ind.

chanical movement to a controlled air signal. Obviously, any moisture control is no more reliable than its sensing device. In this case, the tension roll.

The tension roll should be positioned at the point where the greatest shrinkage and, therefore, the greatest movement takes place. The Stickle Steam Specialties Co., Inc., claims that this point, called the "critical point," is indicated by sheet temperature and is where the sheet temperature suddenly begins to rise rapidly. At this point only 5 to 10% of the water remains to be removed from the sheet. At present our tension rolls are mounted on top of the stack driers, approximately 30 and 36 driers from the dry end.

The tension rolls on both machines have been moved from their original positions, and on one machine the tension roll has been moved several times in order to obtain better sensing results.

FUNCTION OF TENSION ROLLS

The tension roll is connected to a roll arm shaft or rock shaft. The rock shaft, in turn, is connected to a pressure arm on the front side of the machine, which is acted upon by a stabilizer cylinder and an air-loading cylinder. At the back of the machine the rock shaft is connected to a weighted arm which has a moveable weight that rides on it. The movement of the pressure arm is limited by a low- and a high-level stop. The weights on the rock-shaft bar should be adjusted so as to balance the roll and cause the roll, in effect, to have floating action. The rock shaft is a pivot point with the tension roll overhanging it on one side and the weighted arm and stabilizer cylinder to balance it in the floating position, on the other side. This balancing of the tension roll is performed without air on the air-loading cylinder. A cam is connected to the rock shaft and this cam rides against the pilot valve, which gives a modulated signal to the steam valve as the rock shaft rotates. This air signal also is fed to the stabilizer cylinder to minimize oscillations of the tension roll. The tension-roll height is important and affects the amount of force that the sheet can apply to the roll. The cam adjustment is also very critical and changes the linearity of response to movement. Any frictional resistance to movement such as stabilizer clevis binding is detrimental to sensitivity of control. Various sizes of stabilizer cylinders have been used to approach sensitive control and a balanced system without large oscillations.

Failure of Parts

Actual failure of parts has not been a major problem, although the diaphragm in the loading cylinder, because of the temperature at the top of the stacks, has to be replaced every six months or else it deteriorates. This condition is readily detectable and presents no problem. The problem is to receive a reliable signal which varies as the moisture in the sheet varies. Even after following the recommended procedure for balancing the roll and checking the sensitivity of response, 10 p.s.i. at center position, 0 and 20 p.s.i. at the stop points, and after obtaining this response without a sheet on the machine the chart indicated a wide variation in steam pressure and a wandering of the control under operating conditions. At least part of the problem is probably caused by the type of steam valves we have; these are the quick-opening type. The success or failure of a system can depend upon the type of steam valve used. It has been recommended that a V-port valve be used as this valve, supposedly, gives the best results under various loads. I believe there is some merit in new steam valves, but I think that the sensitivity of a response is the other major problem. Because the tension rolls are mounted on the top of the stack driers near the roof, the temperature and moisture conditions make it almost impossible to stay up long enough to make running adjustments to the controls. We are considering moving the tension rolls to a position where they can be adjusted while running.

Effect of Mill Air Supply

Another problem is the mill air supply. It is reduced by Cash Acme valves to 20 p.s.i. for use as instrument air on the Pres-Ten-Trol. However, the mill air supply to these controls is not constant; one air compressor supplies the air to most of the plant, including these controls. The air supply at times is hardly greater than the 20 p.s.i. required and, therefore, the instrument air pressure may vary by 1 to 2 lb. This has very little effect on pressure-control operation, but does adversely affect tension-control operation. One of the pneumatic steam valves closes and the other opens, if the mill air supply fails. This has happened several times either because of compressor difficulties or because the air supply was inadvertently shut off. As a warning against mill air-supply failure, we have purchased a low-air-pressure alarm. The Stickle control has an additional feature, an electric eye, which automatically changes the control from tension- to pressure-operation if the sheet breaks.

Pressure control consists of manually setting the air pressure to the pneumatically operated steam valve. A nozzle flapper arrangement in the control panel tends to maintain a constant steam pressure at the set point. This part of the control has not given any trouble, except for moisture and oil in the air lines.

At present, tension-control operation on both machines is unreliable. The "back tenders" operate the Pres-Ten-Trol on pressure control and adjust the steam pressure to obtain the desired moisture. If machine speed and/or orders are changed, a manual pressure adjustment is required. Operation by pressure control has proved to be reasonably satisfactory but does require, and depend upon, the cooperation and attention of the back tender.

At present the Pres-Ten-Trol is being operated on pressure control for several reasons. Firstly, tension-control operation is uncertain because of a varying air supply which affects operation; secondly, balancing the tension roll for sensitive response is very difficult; thirdly, the type of steam valve does not lend itself to accurate control. Actual mechanical failure of parts has been inconsequential.

Two alternatives are available—either to modify the existing control by replacing the tension roll and its applied equipment, or to use another method and principle of obtaining a more reliable moisture-control signal.

An air compressor is on order because of the inadequate mill air supply. We also intend to install a V-port steam valve for better regulation.

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The Effect of Straining During Drying on the Mechanical and Viscoelastic Behavior of Paper

JOHN H. SCHULZ

A series of handsheets, which had been stretched during drying and maintained in a stretched condition until fully dry, were examined using the Instron load–elongation tester and the tensile creep test. The operation of straining the sheet while wet was termed "wet straining." It was found that wet straining created marked changes in the tensile breaking stress, ultimate elongation, and work-to-rupture of paper. Generally, tensile breaking stress was found to pass through a maximum as wet straining was increased. Response to stress, evaluated by the tensile creep test, was found to pass through a minimum. The elastic modulus, obtained from the initial slope of the load–elongation curves, passed through a maximum as wet straining was increased. No change in the zero-span tensile breaking stress of the sheets was noted. Wet straining was found to reduce the extent of interfiber bonding. It is hypothesized that the observed effects are related changes in the internal structure of the sheet, which affect the distribution of stress in the sheet when supporting a load.

THE EFFECT of stress applied to a sheet during the course of its manufacture has been recognized for some time. Early workers (*1–4*) were largely concerned with the development of tensile strength anisotropy in paper, and they recognized the influence of dry-end variables in that regard. Investigations by Cottral and Gartshore (*5*), Carter (*6*), and Arlov and Ivarsson (*7*) established that tight draws and felts on the paper machine reduce the extensibility of the sheet in the machine direction. Working with laboratory handsheets, Edge (*8*, *9*) found that if a sheet was stretched almost to the breaking point while still wet, the ratio of the machine-direction tensile strength to the cross-machine direction tensile strength would approach a value of four. In this discussion, the term "machine direction" refers to the direction of stress application.

Fujiwara (*10*) concluded that the effect of tension during drying upon the mechanical properties of paper depends upon the degree of wet pressing, the degree of beating, and the method of beating. He found that the amount of tension in the cross-machine direction influences the effect of tension applied simultaneously in the machine direction during drying; but that the influence is not strong and that the essential nature of the machine-direction effects are not altered.

Sapp and Gillespie (*11*), Brecht and Pothman (*12*), and Higgins, Goldsmith, and Harrington (*13*) obtained load–elongation curves for paper dried under various constant loads. It was found that as the stress was increased, the ultimate strength of the dry sheet was increased, but that there is an optimum drying stress beyond which the sheet is weakened by additional loading. Ivarsson (*14*) found no improvement in ultimate strength produced by the application of strain during drying. Briner and Guild (*15*) took samples from different parts of a paper machine and allowed them to dry without further restraint. They concluded that the effect of tension is to weaken paper, although in practice, tension may be necessary to produce a flat sheet. Most of the previous workers agree that drying tensions markedly reduce the extensibility of the finished sheet.

Several authors have presented explanations of the observed effects. The earliest workers associated sheet anisotropy entirely with fiber alignment, and this concept was unchallenged for many years. For example, Robertson and Bailey (*16*) used tensile-strength anisotropy as a measure of fiber orientation, noted that anisotropy developed primarily in the drier section, and, hence, concluded that most of the fibers were oriented in the drier section. However, Danielson and Steenberg (*17*) were able to examine fiber orientation directly, by the observation of tagged fibers. They found that the combing action of the wire was the chief cause of alignment. To evaluate the effect of stretching the wet sheet on fiber orientation, samples were taken from different sections of a paper machine from which the finished sheet was received at a rate 20% greater than the wire speed. Changes in fiber orientation in the drier section were found to be small.

Van den Akker (*18*) has suggested that an important part of the increase in tensile strength caused by drying under tension is the equalization of distribution of stress among the fibers. He points out that when the moisture content is high, the fibers and fiber-to-fiber bonds are in a relatively plastic state and, thus, during drying tension causes fibrous elements between points of bond to become straighter. In that way, all the fibrous elements would partake more equally of the load than if there were no tension. Further, when the paper dries, this condition becomes frozen-in.

While proposing that fiber orientation probably has little effect on this problem, Steenberg (*19*) suggested that the fine fibrils between the fibers may become aligned when the sheet is stretched. He pointed out that this "nap" or fibrillar material is fine enough so that only a very small displacement would be sufficient to cause them to align. This action would change the internal structure of the bonded area, increasing its strength in the direction of tension. Other suggestions concerning the nature of the effect of straining during drying have been made by Berkley and Barker (*20*), and Landt and Rulon (*21*) who proposed that a preferential orientation of the fibrils is involved, and by Corte and Schaschek (*22*), who proposed that the effect

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is related to a preferential orientation of the hydrogen bonds.

THE RESPONSE OF PAPER TO STRESS

The response of paper to stress is dependent upon many factors, some of which are only vaguely understood. Generally, these factors may be divided into three groups: (1) the chemical and physical characteristics of the cellulose and the other material of which the sheet is made, (2) the extent and strength of the bonds which hold the components of the sheet together, and (3) the manner in which stress is distributed among the elements of the sheet when the sheet is in tension.

The first group includes factors such as the degree of polymerization and chemical composition of the cellulose, the degree of crystallization and the size of the crystallites. Factors included in the second group, which involves interfiber bonding, have been reviewed by Swanson (*23*). Hydrogen bonding is presently considered to be the major source of cohesion between the cellulosic components of the sheet. This theory has recently been supported by the work of Corte and Schaschek (*22*). Methods for the measurement of bonded area have been reviewed by Ingmanson and Thode (*24*) and by Swanson and Steber (*25*). Van den Akker (*26*) has discussed the physical structure of a bonded area, and has suggested that the physical condition of the bonded surfaces may strongly affect the structure (and therefore the mechanical properties) of a bonded area. Such differences in structure may make bond strength anisotropic.

The third factor of importance affecting the response of paper to stress is the manner in which stress is distributed within a sheet when supporting a load. Paper is classed as a macroscopically inhomogeneous material. There is great variety in the size, shape, and mechanical properties of the elements which compose the sheet. The least strain resulting from a given load will be realized when the stress in all of these diverse elements is uniformly distributed. The variation in local stress has never been measured. However, it does not seem likely that a very efficient distribution is ever achieved. For structural members generally, abrupt changes in cross section are known to give rise to great irregularities in stress distribution (*27*).

Alfrey (*28*) has discussed the nature of stress distribution in high polymeric materials and suggests it is not correct to assume that a homogeneous macroscopic stress is evenly divided over a given cross-sectional area down to molecular dimensions, even in completely amorphous polymers. In the case of polycrystalline materials, such as cellulose, there is an enormous inhomogeneity of the local stress, even where the macroscopic stress is completely uniform. In paper, stress must be transmitted through the fibers and particles of the sheet, and many opportunities exist for stress inhomogeneity on a macroscopic scale. In addition, the distribution of stress in paper may be complicated by variable local basis weights related to poor formation, and by the different mechanical properties of different components of the sheet. The degree of bonding between the load-supporting elements of the sheet would also seem to be a particularly important factor.

Very little is known of the nature of stress distribution in paper or of how it affects the properties of a sheet. Brezinski (*29*) has obtained evidence that the increase in tensile strength brought about by wet pressing is related to an improved ability to distribute stress evenly among the elements of the sheet. Arlov (*30*) investigated the effect of fiber-length distribution on the load–elongation properties of paper and suggested that the nature of the stress distribution differed among the sheets with differing fiber lengths.

If a material distributes stress in a nonuniform manner among its load-supporting elements, it will be weaker than if the elements all share the stress uniformly. This is because the elements under high stress will fail first and, hence, the load they carried will be redistributed among the remaining elements. It is concluded, then, that stress distribution is intimately involved in both the general prerupture response to stress and the ultimate strength of paper.

The elongation–time–stress relationships of paper have been reviewed by Rance (*31*) and Brezinski (*29*). Brezinski (*29*) examined the tensile creep properties of paper, and observed a degree of ideality in creep response which, he postulated, would not be expected if macroscopic uncurling, straightening, or slipping of the fibers past one another made significant contributions to the total delayed deformation. He suggested that the rate-controlling mechanisms of deformation operate entirely on a molecular level. Early delayed first-creep deformation could be described by a power equation, and later delayed deformation could be described by a logarithmic equation.

The results of Brezinski (*29*), which were largely corroborated in the present study, indicate that the deformation of paper by stress operates primarily on a molecular level, in a manner similar to that which occurs when viscose is subjected to stress. It is evident, however, that this picture is complicated by changes in the macroscopic structure of paper created by stress. In this regard, the increase in bulk of paper under stress reported by Maynard (*32*) and McKee (*33*), and the increase in light-scattering power when under stress, reported by Nordman, Gustafsson, and Olofsson (*34*) and Sanborn (*35*), would seem to be pertinent. These changes in internal structure may involve a time-dependent redistribution of the stress within the sheet when the sheet supports a load.

EXPERIMENTAL PROCEDURES

The purpose of the investigation was to study the effect that straining a wet handsheet has on the mechanical properties of the final, dry sheet. Handsheets were strained immediately after wet pressing and held at constant elongation for the remainder of the drying period. The operation of applying the elongation after wet pressing is called "wet straining." The degree of wet straining (DWS) is defined as the amount of elongation divided by the original sheet length, expressed as a percentage. Only effects produced in the machine direction (the direction of wet straining) were examined, and only positive degrees of wet straining were investigated.

Preparation of Handsheets

The pulp used was a Rayonier, softwood alpha pulp, commercially available as Rayocord G. The pulp was

beaten to four different freenesses with a Valley beater, and at each degree of beating, the pulp was designated by its Schopper-Riegler freeness, expressed in cubic centimeters. For example, pulps 760, 650, 460, and 290 were beaten to Schopper-Riegler freenesses of 760, 650, 460, and 290 cc., respectively. Pulp 760, the least beaten pulp, was further treated to remove the fine material which could pass through a 100-mesh screen on a Clark classifier.

Handsheets were prepared on a 20-in. square sheet mold. A head of water of 54 in. above the wire was used, producing a consistency of about 0.005% to effectively eliminate flocculation, and to improve sheet formation. The sheets were wet pressed to a solids content of 32%.

The apparatus used to wet strain and dry the wet handsheets consisted essentially of a tray of mercury set between two cylinders. The axes of the cylinders were parallel to one another and to the mercury surface. The lowest point of each cylinder was situated parallel to an edge of the tray, and was near and at the same height as the mercury surface. One of the cylinders was stationary, and the other was able to rotate in place.

A sheet to be dried was cut from the wet-pressed web with a razor blade and placed on the mercury surface. The dimensions of the sheet were $4^1/_2$ by 18 in. Each end of the sheet was passed under a cylinder and clamped to that cylinder. A synchronous motor, sufficiently geared down, was used to turn the rotatable cylinder to wet strain the sheet. The sheets were wet strained at the rate of 1% per minute. After the sheet was wet strained the desired amount, the motor was stopped, and the rotatable cylinder was clamped in place until the sheet was fully dried. A series of strain gages attached to the stationary cylinder permitted the recording of the entire stress history of the handsheet during drying, and indicated when straining first began, so that it was possible to control roughly the amount of wet straining applied.

The purpose of the mercury was to support the sheet so that it would not sag or creep under its own weight. The use of the mercury had the additional advantage that the sheet could shrink freely, laterally, during drying.

A series of preliminary experiments indicated that if left to air-dry without restraint from shrinkage in all directions, paper cockles badly. An analysis by Smith (*36*) suggests that cockle can be reduced by insuring a uniform drying rate in all sections of the sheet, so that there are no local moisture gradients to produce different rates of shrinkage in different parts of the sheet. To avoid cockle, the handsheets were dried very slowly. Slow drying permitted sufficient time for the diffusion of moisture through the sheet, so that moisture gradients were largely eliminated. After clamping was accomplished, the unit was enclosed and sealed from the surrounding atmosphere. The surrounding atmosphere was maintained at 50% R.H. and 73°F. and a series of small holes (No. 56 drill) in the cover of the drying unit permitted the high humidity in the vicinity of the sheet in the drying unit to come slowly to equilibrium with the surrounding standard conditions. In this way, the drying rate was slowed, and 192 hr. (8 days) were required to dry the sheets. After eight days, the cover was removed, and after an additional 3-hr. period to insure moisture equilibrium, the sheet was unclamped. This procedure, and the good formation of the sheets, reduced cockle to a minimum.

After wet pressing, the sheets were at 32% solids. The operation of introducing the sheet into the drying unit, which was carefully timed with a stopwatch, took slightly less than 10 min. During this period, the solids content of the sheet was increased by evaporation and, as a result, the solids content of the sheet when it was wet strained was 36%. All the sheets were wet strained at 36% solids.

To determine the degree of wet straining, two dyed fibers were placed about 20 cm. apart on the sheet just before wet pressing. After being pressed into the sheet, the nearest reaches of the dyed fibers were measured. After the sheet had been wet strained, dried, and removed from the drying unit, the distance was measured again, and the difference between the two measurements was used to calculate the DWS.

After removal from the drying unit, the sheet was stored at 50% R.H. and 73°F. All testing was carried out at these standard conditions. Since the sheet was clamped only at its ends, it was free to shrink in its central portion. Because the sheet was long and narrow, shrinkage was uniform in the middle 9 in., and the properties of the sheet in that portion were found to be uniform. All specimens for testing were taken from that middle section.

Bonding Strength Test

The transverse tensile strength test procedure, developed by Eames (*37*) for clay coatings and modified for paper by Jappe and Kaustinen (*38*), was used to provide a measure of bonding strength. The technique essentially involves gripping the two major surfaces and determining the strength of the sheet in the transverse direction (or z-direction). Problems associated with selecting a suitable adhesive and mounting and aligning the specimen have been discussed by Eames (*37*).

The applicability of this approach depends upon the assumption that the strength of paper in the transverse direction depends primarily upon the strength of the bonds between the fibers, and involves little fiber rupture. It should be noted, though, that if the suggestion of Van den Akker (*26*) concerning bond structure mentioned above is correct, the contribution of a bonded area to strength in one sheet direction may bear little relation to the strength in another direction. A suggestion that this situation may be involved in wet straining has been made by Steenberg (*19*).

Tensile Creep Testing

Tensile creep testing involves determining the elongation–time relationship for a specimen supporting constant load. In this investigation, the equipment and techniques developed by Brezinski (*29*) were used. Specimens were 8.25 in. long and 0.87 in. wide. The flat-face clamps used by Brezinski (*29*) were found to exhibit partial pull-out amounting to several per cent of the total creep, and, for most of the tests, a set of line-contact clamps, similar to those suggested by Van den Akker and Hardacker (*39*) and Sanborn and Diaz (*40*), were used.

Table I. The Mechanical Properties of Handsheets Subjected to Different Degrees of Wet Straining

Pulp No.	*Degree of wet straining, %*	*Tensile breaking stress, kg./sq. mm.*	*Ultimate elongation, %*	*Work-to-rupture, kg.-cm./cc.*	*Basis weight, g.s.m.*
760	1.2	4.51	2.44	8.75	66.6
	2.1	4.51	2.44	9.02	63.3
	2.2	4.34	2.28	7.72	60.0
	2.3	4.46	2.62	9.18	64.7
	3.1	4.64	2.38	8.40	62.4
	4.5	4.89	1.93	7.03	63.4
	5.0	5.10	1.63	6.07	66.6
	6.1	4.74	1.45	4.94	64.3
	6.3	4.68	1.54	5.39	62.1
650	0.0	6.72	3.12	15.68	65.0
	0.9	7.13	2.75	14.70	65.6
	1.6	7.42	2.75	15.60	65.5
	2.2	7.52	2.50	14.35	64.2
	2.3	7.57	2.32	12.16	85.0
	2.8	7.64	2.25	12.38	64.1
	3.5	7.80	2.18	9.76	84.8
	4.5	7.80	2.06	10.80	84.7
	5.1	7.63	2.00	10.82	84.7
	5.9	7.30	1.46	7.84	64.0
	6.0	7.28	1.63	8.18	83.2
460	0.7	5.13	2.04	7.94	72.3
	1.8	5.64	2.01	8.56	69.1
	2.1	5.59	2.01	8.46	67.4
	3.4	6.21	1.93	8.85	67.4
	4.2	6.75	2.12	9.60	67.0
	4.7	7.17	1.83	9.60	69.1
	6.3	7.15	1.81	9.30	66.0
	6.7	6.58	1.83	8.80	66.0
290	1.0	6.31	1.89	8.97	87.6
	1.9	6.53	2.12	10.43	85.0
	2.4	6.74	1.68	8.35	79.6
	4.0	7.21	1.94	10.29	70.9
	4.6	7.22	1.76	9.14	81.5
	4.9	6.98	...	...	82.1
	5.1	7.42	1.38	7.10	77.0
	6.6	7.90	1.64	9.25	80.7

Creep tests are described in part by the "apparent initial stress" to which the specimen is subjected. The apparent initial stress is determined by dividing the applied load plus the weight of the lower clamp (300 g.) by the calculated cross-sectional area of the cellulose supporting the load. The area involved is perpendicular to the direction of the applied load. It may be calculated after accurately determining the weight and length of the specimen, and assuming the density of airdry cellulose to be 1.55 g./cc. The stress is termed "apparent" because of the great variations in local stress which, presumably, are present in paper under tension. The apparent initial stress is given in terms of kg./sq. mm.

EXPERIMENTAL RESULTS

Load–Elongation Tests

The load–elongation properties of the handsheets were determined using the Instron load–elongation tester. The applicability of the Instron instrument for this purpose has been examined by Van den Akker and Hardacker (*39*). Testing was done at 50% R.H. and 73°F. The samples were strained in the Instron at the rate of 6.2% per minute. The information obtained from the load–elongation curves is presented in Table I for handsheets of different degrees of wet straining. Included in this table are ultimate strength, ultimate elongation, and the amount of work necessary to cause the failure of the specimen.

As the DWS was increased, it was observed generally that the tensile breaking stress increased, passed through a maximum, and then decreased. This is illustrated in Fig. 1 for pulp 650. In this case, the maximum tensile breaking stress was reached at DWS of about 3.5%. The largest increase was observed with pulp 460, in which case the highest stress recorded was about 40% greater than the tensile breaking stress of the sheet prepared with the lowest degree of wet straining. The tensile breaking stress of pulp 290 increased continuously with increased wet straining, and no maximum was observed. It is suggested, however, that higher degrees of wet straining might have produced a maximum in the stress.

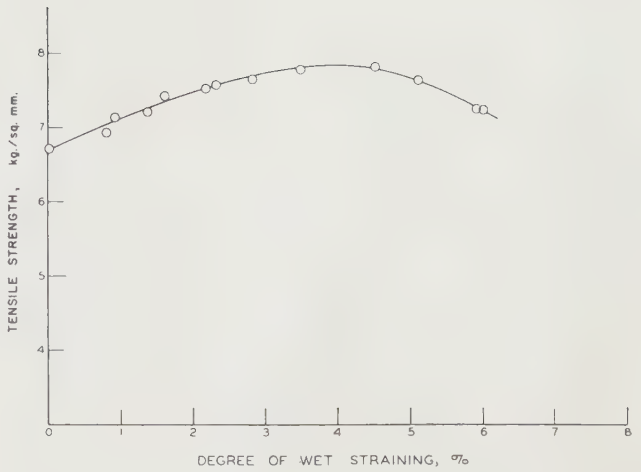

Fig. 1. The effect of wet straining on tensile breaking stress for pulp 650

The effect of wet straining on ultimate elongation and work-to-rupture was found to be strongly dependent upon the degree of beating. For pulps 760 and 650, ultimate elongation and work-to-rupture were found to drop markedly as the DWS was increased. This effect is shown in Fig. 2 and 3 for the ultimate elongation of pulp 760 and the work-to-rupture of pulp 650, respectively. The work-to-rupture of pulp 460 passed through a maximum with increased wet straining, as shown in Fig. 4. A slight drop in ultimate elongation was noted as the DWS was increased using pulps 460 and 290. It should be noted that the ultimate elongation and work-to-rupture data for pulp 290 exhibited scatter which may be related to the poorer formation of this slower draining pulp.

The remainder of the experimental program was concerned largely with examining the changes in sheet properties, produced by wet straining, which were responsible for the changes in load–elongation characteristics noted above. Attention was focused upon the intrinsic strength of the cellulose, the bonding characteristics of the sheet, and the response of the sheet to stress as evaluated by the tensile creep test.

Intrinsic Fiber Strength

Wet straining has long been associated with preferred fiber orientations in the sheet. The theory proposes that when the sheet is wet-strained, the fibers tend to become aligned in the direction of the applied stress. The effects of wet straining are attributed to this alignment. Proponents point out that as the sheet strength increases in the machine direction, cross-machine-direction strength is often observed to decrease.

The work of Danielson and Steenberg (*17*), noted above, would indicate that the effects of wet straining

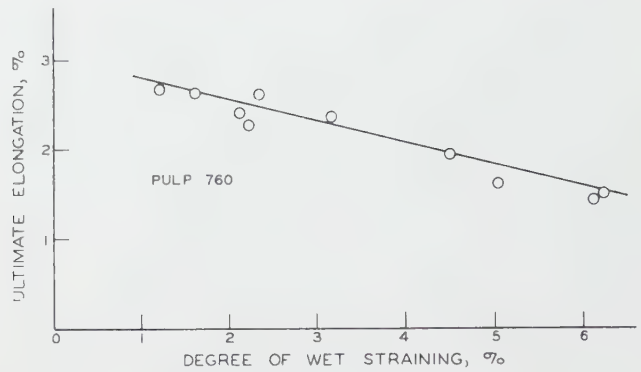

Fig. 2. The effect of wet straining on the ultimate elongation of pulp 760

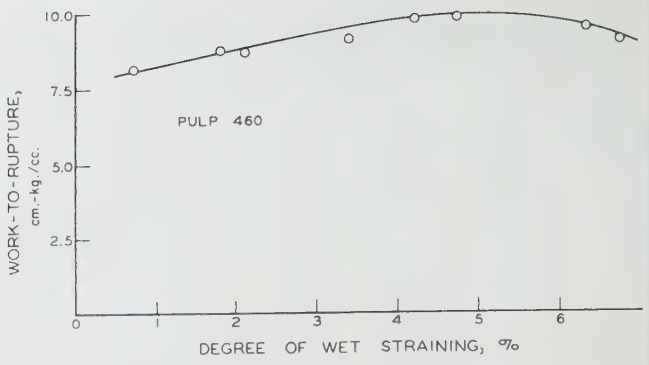

Fig. 4. The effect of wet straining on the work-to-rupture of pulp 460

are not related to the development of preferred fiber orientations. Therefore, a detailed examination of fiber orientations was not undertaken. However, it is hypothesized that if the fibers are oriented by wet straining, an increase in the zero-span tensile breaking stress of the sheet in the direction of wet straining would be noted. This hypothesis follows from an analysis of the zero-span tensile strength test developed by Van den Akker, Lathrop, Voelker, and Dearth (*41*).

Zero-span clamping jaws, improved by Wink (*42*) at The Institute of Paper Chemistry, were used to examine the zero-span tensile breaking stress of handsheets prepared with different degrees of wet straining. The results are shown in Table II. At each degree of beating, the hypothesis of equal zero-span tensile breaking stresses for all degrees of wet straining falls well within 95% confidence limits. It is concluded that wet straining does not increase the tensile breaking stress of the fibers in the direction of straining, and therefore it is not likely that gross fiber realignment or fibril orientation are related to the wet-straining effect. This does not mean that fibril orientation does not take place. Rather, it is an indication that even if such orientation occurs, its effect is probably of minor importance.

Bonding Characteristics

It has been found that small amounts of wet straining act to increase the tensile strength of paper. This increase could be due to an effect on the strength of the bonds between the fibers. Stronger interfiber bonding may be the result of an increase in the actual intrinsic strength of the bonds, or an increase in the relative bonded area. In either case, the total strength of the bonds would be increased. In this study, bonding effectiveness was examined using the transverse tensile strength test and the light-scattering characteristics of the handsheets.

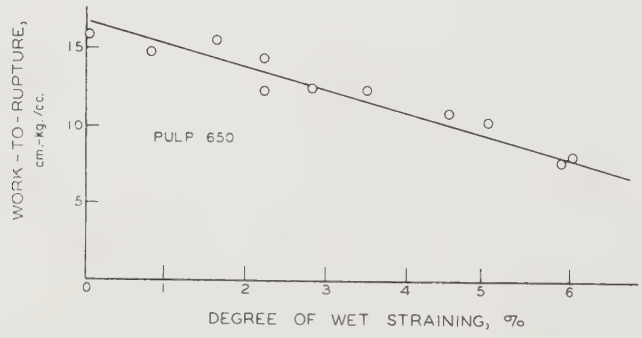

Fig. 3. The effect of wet straining on the work-to-rupture for pulp 650

The transverse tensile strengths of handsheets prepared with different degrees of wet straining from pulps 650, 460, and 290 are presented in Table III. A similar investigation of handsheets prepared from pulp 760 was not carried out because of penetration of this more porous pulp by the adhesive. This difficulty might have been overcome by increasing the basis weight of the sheets prepared from this pulp. No attempt was made to do this, however, because it was felt that the three suitable pulps available reasonably represent the nature of the effect.

The results are reported in terms of kilograms per square millimeter. The force is that necessary to separate the two lugs attached to either side of the paper sample. The area term refers to the measured planar area of the faces of the lugs, or alternately, to the measured planar area of the tested sample. It bears no relation to the actual area created by the rupturing of the bonds in the sheet. If this actual area were known, then it would be possible to have a true measure of the intrinsic bonding strength. The value obtained should be regarded as a measure of the force necessary to cleave the sheet in a plane parallel to the surfaces of the sheet, and is thus an indication of the relative total strength of the bonds between the fibers.

As the degree of wet straining is increased, the transverse tensile breaking stress of the sheets decreases, indicating that wet straining reduces the bonding strength of paper. This is concluded from the results of tests using handsheets prepared from all three pulps examined in this manner. It is noted that, as the degree of beating is increased, the percentage drop in transverse tensile breaking stress decreases. This effect may be related to the increased conformability and decreased fiber length of the more refined pulp.

The light-scattering characteristics of paper have been reviewed recently by Ingmanson and Thode (*24*). The ability to scatter light, characterized by a scattering coefficient, is assumed to be directly proportional to the unbonded fiber area, since the fiber surface involved in bonding does not scatter light. Therefore, the scattering coefficient is used as a measure of the unbonded fiber specific surface.

The scattering coefficients of handsheets prepared from pulps 760 and 650 were determined using a General Electric recording spectrophotometer at an effective wavelength of 650 mμ. It was found that, as the degree of wet straining was increased, the scattering

Table II. The Zero-Span Tensile Breaking Stress of Sheets Subjected to Different Degrees of Wet Straining

Pulp No.	*Degree of wet straining, %*	*Zero-span tensile breaking stress kg./sq. mm.*
760	1.4	34.4
	2.2	34.0
	3.1	34.5
	5.0	34.2
	6.3	34.9
290	1.0	31.7
	2.4	33.2
	4.9	31.6
	6.6	33.2
650	1.4	40.0
	2.6	42.7
	2.9	40.7
	5.4	40.9
	6.3	42.1

coefficients for both of these pulps increased, indicating a decrease in the optical contact between the fibers of the sheet. This behavior is shown in Fig. 5 for pulp 650. Scattering coefficients for pulps 460 and 290 were not determined because of contamination of the surfaces of the sheets through contact with the mercury during their preparation.

It is emphasized that all wet straining took place at 36% solids. The work of Lyne and Gallay (*43*) would indicate that, at this solids content, there is only meager bonding between the fibers and that this bonding accounts for only a small part of the total bonding in the dried sheet. It does not seem likely that the breaking of a part, or even all, of the bonds present at 36% solids accounts entirely for the decrease in bonded area observed in these sheets. Even if many of these early-formed bonds are broken, they may be able to reform during a later stage of the drying period.

Rather, it seems more likely that during wet straining, part of the potential for bonding during the remainder of the drying period is destroyed. It is suggested that this may occur if wet straining changes the proximity of the fibers to one another, so they are not in an advantageous position for bonding. During wet pressing, the potential for interfiber bonding is improved if the fibers are made to partially wrap or conform around one another, so they are in a good position to be drawn together by the Campbell effect (*44*). The increased scattering coefficients noted in Fig. 5, and the decreased transverse tensile breaking stress noted

Table III. The Effect of Wet Straining on the Transverse Tensile Breaking Stress

Pulp No.	*Degree of wet straining, %*	*Transverse tensile breaking stress, kg./sq. mm.*
650	0.8	0.162 (231)
	1.4	0.158 (224)
	2.8	0.142 (202)
	4.9	0.133 (189)
	5.1	0.120 (170)
	6.0	0.118 (168)
460	0.9	0.248 (352)
	1.2	0.249 (354)
	2.1	0.250 (355)
	4.7	0.237 (337)
	5.8	0.224 (318)
	6.7	0.224 (318)
290	1.1	0.412 (586)
	2.4	0.405 (576)
	4.0	0.391 (556)
	5.1	0.387 (551)

Note: The figures in parentheses are in quivalent units of p.s.i.

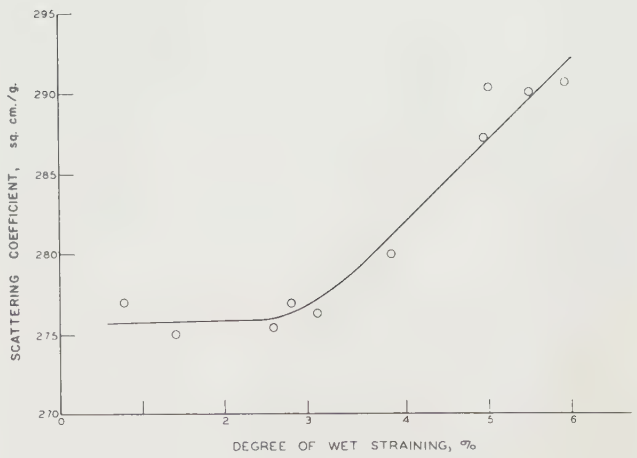

Fig. 5. The effect of wet straining on the light-scattering coefficient of pulp 650

in Table III, would be expected if wet straining undid some of this conforming action. Perhaps some of the coherence of the sheet produced by wet pressing is destroyed by wet straining, and the dried sheet is then less well bonded.

The data presented here suggest that increased bonding between the fibers of the sheet does not account for the increase in tensile breaking stress noted in Fig. 1. However, it may be hypothesized that some other mechanism acts to increase the tensile breaking stress up to the maximum and then becomes less effective as wet straining is increased. The observed drop in tensile breaking stress at higher degrees of wet straining may be related to the drop in bonding effectiveness noted here.

First Creep

The first time a specimen is subjected to a constant load in an elongation–time test is called the *first creep test.* If, after some time, the load is removed and a period of recovery is allowed, followed by the reapplication of the load, the second period during which the specimen is under load is called the *second creep test,* and so on. Similarly, the period of recovery immediately following the first creep test is called the *first recovery test.*

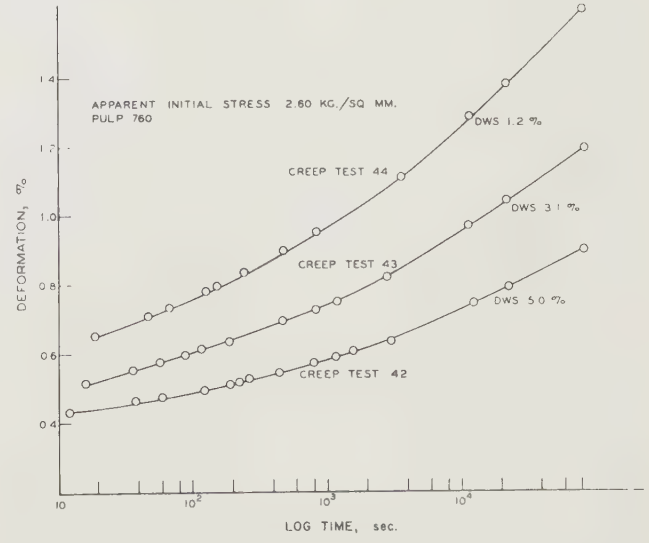

Fig. 6. First-creep curves for handsheets of different degrees of wet straining

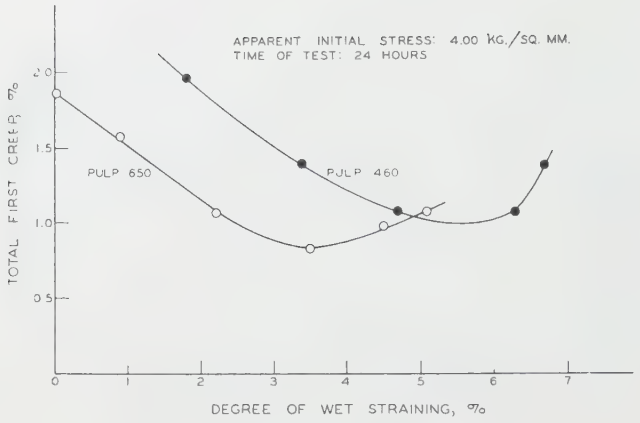

Fig. 7. The effect of wet straining on total first creep

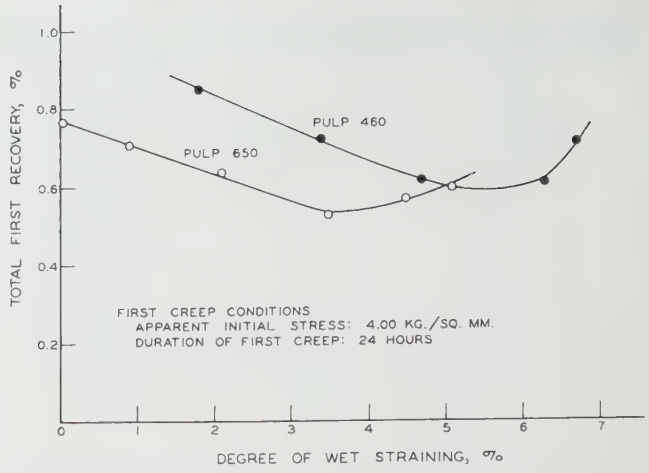
PULP 460
PULP 650
FIRST CREEP CONDITIONS
APPARENT INITIAL STRESS: 4.00 KG./SQ. MM.
DURATION OF FIRST CREEP: 24 HOURS
TOTAL FIRST RECOVERY, %
DEGREE OF WET STRAINING, %

Fig. 8. The effect of wet straining on total first recovery

Several first-creep curves for handsheets prepared from pulp 760 with different degrees of wet straining are shown in Fig. 6. These curves are prepared by plotting the deformation produced by the load, expressed as a percentage of the original sheet length, against the logarithm of time, expressed in seconds. In Fig. 6, the apparent initial stress was 2.60 kg./sq. mm. in all cases. It is seen that at the same apparent initial stress, distinct differences exist between first creep curves obtained using handsheets with different degrees of wet straining.

This effect may be examined more closely in Fig. 7, where the total first creep in 24 hr. produced by an apparent initial stress of 4.00 kg./sq. mm. for handsheets prepared from pulps 650 and 460 is shown as a function of DWS. It is seen that as the DWS is increased, the total first creep, which includes the immediate elastic deformation and the delayed deformation, decreases, passes through a minimum, and then increases. Similar behavior was observed in the delayed deformation between any two arbitrarily chosen times in these first creep tests. It is noted that the minimum in first-creep response corresponds to the DWS at which the tensile strength was a maximum.

It was found that first creep curves for any single handsheet at any DWS could be fitted into a master creep curve using the method of Brezinski (*29*).

If the changes brought about in first creep by wet straining are compared with the changes in bonding characteristics noted earlier, an interesting phenomenon is revealed. The drop in transverse tensile breaking stress and the increase in scattering coefficients indicate that as wet straining is increased, the bonding between the components of the sheet becomes progressively diminished. However, it is found concurrently that the prerupture response of the paper to stress is generally diminished with increased wet straining. In other words, sheets with less well-developed bonds between their fibers are able to exhibit less creep than some sheets with greater bonding, made from the same pulp. This is particularly well documented for pulp 650. It is meant to point out by this that bonding characteristics constitute only one factor among several which govern prerupture response, and bonding may not be the most important factor in many cases. This evidence should cast doubt on any theories of prerupture response to stress which depend primarily upon fiber slippage or unkinking for the rate-controlling mechanism.

Recoverable Deformation

It has been seen that wet straining creates marked changes in the extent of first creep under comparable apparent initial stresses. The possibility exists, though, that these differences are due to differences in the capacity of the sheets for nonrecoverable deformation. If such is the case, the effect of wet straining could be called one of mechanical conditioning. Therefore, it was important to determine if differences in recoverable deformation are created by wet straining.

One measure of creep by recoverable mechanisms is the total first recovery following first creep. The effect of wet straining on total first recovery in 24 hr. for handsheets prepared from pulps 650 and 460 is shown in Fig. 8. These recovery tests followed 24-hr. first-creep tests at an apparent initial stress of 4.00 kg./sq. mm. The weight of the clamps remained on the specimens during the recovery period. It is seen that the effect is similar to the effect on total first creep, shown in Fig. 6.

Another approach to this problem would be to measure creep after the nonrecoverable deformation has been removed. Nonrecoverable deformation was removed by subjecting the specimens to a series of multiple cycle tests, in which the same apparent initial stress was applied to the specimen and removed many times. The periods of creep and the periods of recovery in these tests were equal in length. After many cycles, when most of the nonrecoverable deformation has been removed, the specimens are said to be mechanically conditioned to stress.

It was found that even after 8 or 9 cycles in such tests, large differences still exist in the response to stress of specimens subjected to different degrees of wet straining. The effect is illustrated in Table IV where the total fourth creep for handsheets prepared from pulp 650 with different degrees of wet straining is presented.

Table IV. Total Fourth Creep Deformation at Different Degrees of Wet Straining (DWS) for Pulp 650

DWS, %	*Total creep deformation, %*
0.0	0.740
0.9	0.680
3.5	0.524
4.5	0.537
5.1	0.564

Note: Apparent initial stress = 4.00 kg./sq. mm. Time: 24 hr.

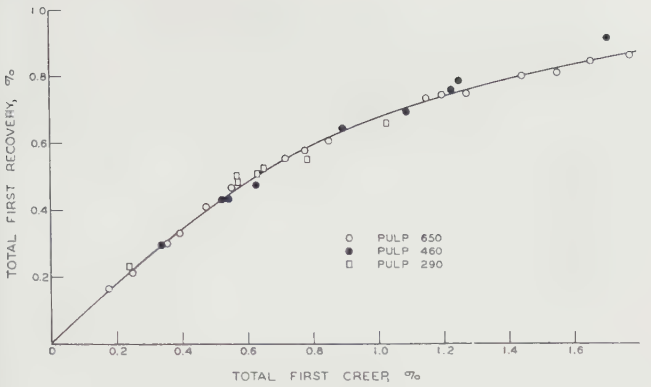

Fig. 9. The relationship between total first creep and total first recovery

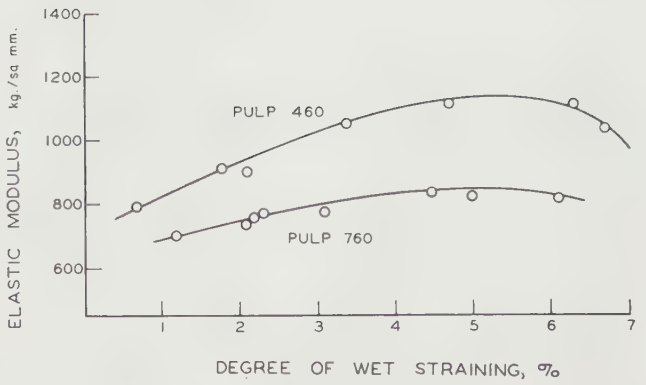

Fig. 10. The effect of straining on the elastic modulus of pulps 460 and 760

Since, in the fourth creep test, almost all of the capacity for deformation by nonrecoverable mechanisms has been removed, it must be concluded that wet straining affects the capacity for creep by recoverable mechanisms. Table IV shows that the total fourth creep passes through a minimum in the same manner as the total first creep and the total first recovery.

It may be the case, however, that in addition to the change in creep capacity by recoverable mechanisms, wet straining may affect the capacity for creep by nonrecoverable mechanisms. Because of the change in recoverable creep, though, demonstration of such an effect is very difficult. One approach is to plot, for specimens taken from handsheets with different degrees of wet straining, total first creep versus total first recovery, for constant, equal periods of creep and recovery. If the first creep–first recovery relationship is determined for a handsheet with no wet straining, the resulting curve would be expected to lie lower than the first creep–first recovery relationship for a handsheet from which some of the capacity for nonrecoverable deformation has been removed. In the latter handsheet, creep would be more nearly equal to recovery in extent.

When this relationship was determined using different apparent initial stresses on handsheets prepared from pulps 650, 460, and 290 with different degrees of wet straining, it was found that a single curve could be drawn through all of the datum points. In other words, a unique relationship was established between first creep and first recovery which was independent of DWS and stress, and which was not strongly affected by beating. Further it was found that the time of the test did not affect the relationship, as long as the time of recovery was equal in length to the period of creep.

This relationship is presented in Fig. 9. A single curve is drawn through all of the points to emphasize the similar behavior of all three pulps tested. However, smooth curves drawn through the data obtained at any one particular degree of beating are not quite coincident. These data were obtained using the line-contact clamps. No load remained on the specimen during the recovery period.

The unique relationship between first creep and first recovery for sheets prepared with different degrees of wet straining indicates that the amount of recovery is related only to the first-creep deformation, and is not affected by wet straining. This would suggest that the capacity of the sheet for creep by nonrecoverable mechanisms is not affected by wet straining. Therefore, the effects of wet straining should not be considered to operate by mechanisms which are important to the mechanical conditioning of paper.

Apparent Elastic Modulus

It would be of value to examine the response to stress of wet-strained paper, using a measure which is not time dependent. Among the time independent mechanical properties of paper is the *elastic modulus*. Paper is not an elastic material. Brezinski (*29*) has shown that even under very low loads, the deformation of paper is time dependent. However, it was demonstrated that the extent of time-dependent deformation is stress proportional, and at low loads the rate of time-dependent deformation is low. Because of this, it is often possible to obtain a valid estimate of the elastic modulus of paper by measuring the initial slope of the load–elongation curve. This procedure is based on the assumption that during the early part of the load–elongation test, the rate of elongation is large compared to the rate of creep.

The change in apparent elastic modulus brought about by wet straining is shown in Fig. 10 for handsheets made from pulps 760 and 460. These data were obtained by measuring the initial slope of the load–elongation curves. It is preferred to refer to these values as *apparent* elastic moduli because of the difficulty, previously noted, in rigorously defining stress. The units of the moduli are given as kilograms per square millimeter and are presented as the apparent stress per unit strain. The strain is calculated as the elongation of the specimen divided by the specimen length.

It is seen that as the DWS is increased, the moduli increase, pass through a maximum, and then decrease. Similar behavior was observed in the elastic moduli of handsheets prepared from pulp 650. The maxima correspond to the DWS at which maximum tensile strength, minimum first creep, and minimum first recovery were noted. It was found that as the DWS was increased, the elastic moduli of handsheets prepared from pulp 290 increased continuously.

CONCLUSIONS

It is hypothesized that wet straining affects the manner in which stress is distributed among the elements of the sheet when the sheet supports a load. When a load is applied to paper, the stress developed is distributed in a nonuniform manner within the sheet. If this stress distribution is altered, the measured response of the sheet to stress will be affected. It is proposed that the observed changes in first creep, recover-

able creep, and elastic modulus, are related to a changed distribution of stress in the sheet.

As the DWS was increased, the creep response passed through a minimum, and the elastic modulus passed through a maximum. It is suggested that wet straining caused the creep response to fall by creating a more uniform distribution of stress among the elements of the sheet, so that more of the fibrous elements shared in supporting the load. This effect also caused the tensile breaking stress to become greater. Regarding the general effect on viscoelastic behavior, the sheets with the more uniform stress distribution responded at any time in a manner characteristic of response at an earlier time for sheets with a less uniform stress distribution.

The basic mechanism whereby the stress distribution in the sheet is changed is not known and cannot be described with certainty at the present time. It is hypothesized that when the sheet is wet strained, the fibers mainly slide past one another, although some hydrogen-bond breaking may occur. This sliding or slipping action would seem to cause some of the fibrous elements to assume slightly different relative positions which, when the sheet is dry, would be more effective in contributing to the support of a load. This action may involve the straightening of bends and kinks between points of bonding. Because the zero-span tensile breaking stress apparently was unaffected by wet straining, gross fiber realignment probably is not involved. After the sheet has dried, it would be expected to be stronger and to deform less under stress applied in the direction of wet straining.

The hypothesized mechanical actions are consistent with the finding that the bonding effectiveness of the fibers is decreased by wet straining. Apparently, the sliding and straightening of the fiber elements destroys some of the coherence of the sheet, and the ability of the sheet to form interfiber bonds during the remainder of the drying period is impaired. The weakening of the sheet by this behavior is apparently overcome by the improvement in stress distribution created by small amounts of wet straining, since it is found that small amounts of wet straining cause the tensile strength of the sheet to increase.

The observed changes in creep response and elastic modulus suggest that small amounts of wet straining tend to improve the stress distribution, but that this improvement is halted and the effect is reversed as the action is continued. This reversal is probably related to the disruption of the sheet noted above. The hypothesized disruption could produce both the decrease in bonded area and the tendency for the stress distribution to become less uniform, and would account for the drop in tensile breaking stress at higher degrees of wet straining which was observed.

LITERATURE CITED

1. Rieth, E., *Papier-Fabr.* 27: 385 (1929).
2. Haury, F., *Papier-Fabr.* 66: 515 (1935).
3. Adams, F. W., *Paper Trade J.* 101, No. 24: 33–35 (Dec. 12, 1935).
4. Lindman, B. P., *Papir-J.* 27, No. 16: 246–252 (Aug. 31–Sept. 6, 1939); *B.I.P.C.* 10: 82.
5. Cottral, L. G. and Gartshore, J. L., *Brit. Paper and Board Makers' Assoc., Proc. Tech. Sect.* 24: 261–307 (1943).
6. Carter W. J., *Brit. Paper and Board Makers' Assoc., Proc. Tech. Sect.* 25: 231–233 (1944).
7. Arlov, A. P. and Ivarsson, B., *Svensk Papperstidn.* 54, No. 21: 729–738 (Nov., 1951).
8. Edge, S. R. H., *Brit. Paper and Board Makers' Assoc., Proc. Tech. Sect.* 25: 210–215 (1944).
9. Edge, S. R. H., *Chem. & Ind.* (London) 67, No. 51: 803–807 (Dec., 1948).
10. Fujiwara, Y., *J. Japan. Tech. Assoc. Pulp Paper Ind.* 11, No. 4: 30–33 (1957).
11. Sapp, J. E. and Gillespie, W. F., *Paper Trade J.* 124, No. 9: 120, 122, 124, 126 (Feb. 27, 1947).
12. Brecht, W. and Pothmann, D., *Das Papier* 9, No. 13/14: 304–311 (July–Sept., 1955).
13. Higgins, H. G., Goldsmith, V., and Harrington, K. J., *Australian J. Appl. Sci.* 6, No. 4: 486–510 (Dec., 1955).
14. Ivarsson, B. W., *Tappi* 37, No. 12: 634–639 (Dec., 1954).
15. Briner, E. A. and Guild, H. J., U. S. pat. 1,573,297 (Feb. 16, 1926).
16. Robertson, J. A. and Bailey, F. W., *Brit. Paper and Board Makers' Assoc., Proc. Tech. Sect.* 15, No. 1: 155–159 (1934).
17. Danielson, R. and Steenberg, B., *Svensk Papperstidn.* 50, No. 13: 301–305 (Nov., 1955).
18. Van den Akker, J. A., *Tappi* 33, No. 8: 398–402 (Aug., 1950).
19. Steenberg, B., *Finnish Paper Timber J.* 29, No. 7A: 64–69 (1947).
20. Berkley, E. E. and Barker, H. D., U. S. Dept. Agr. Tech. Bull. No. 949, p. 21, Washington, U. S. Gov't. Printing Office, 1948.
21. Landt, G. E. and Rulon, S. A., *Paper Trade J.* 111, No. 4: 32–36 (July 25, 1940).
22. Corte, H. and Schaschek, H., *Das Papier* 9, No. 21/22: 519–530 (Nov., 1955).
23. Swanson, J. W., *Tappi* 39, No. 5: 257–270 (May, 1956).
24. Ingmanson, W. L. and Thode, E. F., *Tappi* 42, No. 1: 83–93 (Jan., 1959).
25. Swanson, J. W. and Steber, A., *Tappi* 42, No. 12: 986–994 (Dec., 1959).
26. Van den Akker, J. A., *Tappi* 42, No. 12: 940–947 (Dec., 1959).
27. Timoshenko, S., "Strength of materials," 3rd ed., New York, Van Nostrand, 1955.
28. Alfrey, T., "Mechanical behavior of high polymers," New York, Interscience, 1948. 581 p.
29. Brezinski, J. P., "A study of the viscoelastic properties of paper by means of tensile creep tests." Doctor's Dissertation, Appleton, Wis., The Institute of Paper Chemistry, 1955. 242 p.; *Tappi* 39, No. 2: 116–128 (Feb., 1958).
30. Arlov, A. P., *Pulp Paper Mag. Can.* 53, No. 1: 84–91 (Jan., 1952).
31. Rance, H. F., "The mechanical properties of paper," *In* Meredith's Mechanical properties of wood and paper, New York, Interscience, 1953.
32. Maynard, C. R. G., *Brit. Paper and Board Makers' Assoc., Proc. Tech. Sect.* 29: 470 (1948).
33. McKee, R. C., Private communication, The Institute of Paper Chemistry, 1958.
34. Nordman, L., Gustafsson, C., and Olofsson, G., *Paper and Timber* (Finland) 36, No. 8: 315–320 (1954).
35. Sanborn, I. B., Personal communication, The Institute of Paper Chemistry, 1959.
36. Smith, S. F., *Brit. Paper and Board Makers' Assoc., Proc. Tech. Sect.* 31, No. 1: 107–126 (1950).
37. Eames, A. C., "The transverse tensile strength of clay-starch coatings as a function of adhesive distribution," Doctor's Dissertation, Appleton, Wis., The Institute of Paper Chemistry, 1959. 135 p.
38. Jappe, N. and Kaustinen, O., Unpublished work, The Institute of Paper Chemistry, 1958.
39. Van den Akker, J. A. and Hardacker, K. W., *Tappi* 41, No. 8: 224–231A (Aug., 1958).
40. Sanborn, I. B. and Diaz, R. J., *Tappi* 42, No. 7: 588–597 (July, 1959).
41. Van den Akker, J. A., Lathrop, A. L., Voelker, M. H., and Dearth, L. R., *Tappi* 41, No. 8: 416–425 (Aug., 1958).
42. Wink, W., Unpublished work, The Institute of Paper Chemistry, 1960.
43. Lyne, L. M. and Gallay, W., *Tappi* 37, No. 12: 581–586, 694–704 (Dec., 1954).
44. Campbell, W. B., "The cellulose-water relationship in papermaking," Forest Prods. Lab. of Canada, Ottawa, 1933, 52 p. (Can. Dept. of the Interior Forest Service Bull, 84).

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The Determination of Pentosans by Ultraviolet Absorption Spectrophotometry

HAROLD L. JONES

A precise and relatively simple method for determining the pentosans content of wood pulps is based on ultraviolet absorption spectrophotometry. A correction for hydroxymethylfurfural is applied in the course of each determination. This is accomplished by collecting a volume of distillate in excess of that required for the estimation of furfural. The additional distillate is used in the reference cell of the spectrophotometer during the measurement of absorbence of the corresponding furfural solution. Since the rate of distillation of hydroxymethylfurfural is constant under controlled conditions, absorbence at the selected wavelength serves as a measure of the furfural content of the analytical solution.

The determination of pentosans in wood pulps is commonly carried out in two steps. The first is the hydrolysis of pentosans to produce pentoses, and ultimately furfural; the second is the estimation of the furfural resulting from these reactions. Hydrolysis is usually effected by the action of boiling hydrochloric acid. During the course of the reaction furfural is distilled from the mixture and is recovered in the distillate.

COMPLICATING FACTORS

Although many methods are available for the determination of furfural—some of them quite accurate in the absence of interfering materials—the problem of the estimation of pentosans is complicated by the following circumstances:

1. A significant part of furfural is always lost during distillation. Adams and Castagne (*1*) report 90% of theoretical recovery during simple hydrolysis of pentosans with 12% HCl saturated with sodium chloride.

2. Other materials besides pentosans—uronic acids, polyuronides, and oxycellulose—yield furfural when treated with hydrochloric acid under the conditions of the test.

3. Other materials besides furfural—hydroxymethylfurfural from hexoses and hexosans, and methylfurfural from methylpentoses and methylpentosans—are formed when pulp is digested with boiling hydrochloric acid.

The recovery of furfural by distillation is probably as nearly quantitative under the conditions described in method CCA 24 of the Swedish Association of Pulp and Paper Engineers (*2*) as by the application of any of the many schemes that have been suggested by investigators in this field.

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The formation of furfural from materials other than pentosans may be the source of serious error, especially in the analysis of pulps of high polyuronide content. Browning (*3*) suggests that the total furfural yield may be corrected for the amount of furfural that corresponds to the carbon dioxide found in the uronic acid analysis. The application of this correction is time-consuming, however, and although the error is recognized, usually no attempt is made to compensate for it.

Wilson and Mandel (*4*) list as the principal methods for the determination of furfural: (*1*) precipitation with phloroglucinol or barbituric acid, (*2*) colorimetric estimation by the use of orcinol or aniline acetate, (*3*) ultraviolet absorption spectrophotometry, and (*4*) bromination. Hydroxymethylfurfural is reported to interfere in each of these analyses, except in those depending upon the development of color with orcinol or aniline. Even so, the possible influence of hydroxymethylfurfural on the development of color with aniline is not completely ruled out. The ultraviolet spectra of furfural and hydroxymethylfurfural are so similar (bands of maximum absorption are at 278 and 284 mμ, respectively) that Wilson and Mandel dismissed ultraviolet absorption spectrophotometry from serious consideration as a useful method of analysis, except as it might serve in mill laboratories as the basis for quick control tests (*4*).

Correcting for Hydroxymethylfurfural

In attempting to confirm the observations of Launer and Wilson (*5*) relative to the uniformity of rate of hydroxymethylfurfural from cellulose, Johansson (*6*) showed that by collecting a separate sample of distillate, in addition to that considered adequate for the determination of furfural, a means is provided for correcting for the presence of hydroxymethylfurfural. If the rate of distillation is kept constant through the entire operation, the concentration of hydroxymethylfurfural in the second sample will be the same as in the first. Thus the extra distillate may be used as a reference solution in the spectrophotometric determination of furfural. Correction for the presence of hydroxymethylfurfural will be made automatically without further manipulation. Johansson applied the extra distillate technique to the determination of pentosans in which color is developed by interaction of furfural and orcinol.

Smith and Rogers (*7*, *8*) described a "highly sensitive" method for analyzing for furfural plus hydroxymethylfurfural by means of ultraviolet absorption. Using the aniline acetate method of Stillings and Browning (*9*) for determining furfural, and ultraviolet absorption for furfural plus hydroxymethylfurfural they calculated the hydroxymethylfurfural content of the

distillate by difference. In a series of factorial experiments it was again shown that under uniformly controlled conditions the rate of formation of hydroxymethylfurfural from cellulose is constant; that it is independent of the type of pulp under examination. Smith and Rogers (7) made this circumstance the basis for the establishment of a correction factor to be used in calculating the pentosans content of pulp samples from ultraviolet absorption characteristics of the distillate.

Table I. Comparison of Data from Ultraviolet Absorption, Orcinol, and Bromate Methods of Analysis of Pulp

Pulp sample	UV Absorption (277 mμ) Distillate for reference		Pentosans, % Orcinol method Distillate for reference		Bromate method[a]	
I	0.41				0.24	
	0.36				0.23	
		0.38	...			0.24
II	1.45				1.22	
	1.45				1.20	
		1.45	...			1.21
III	1.73		2.17		1.66	
	1.65		2.00		1.62	
		1.69		2.08		1.64
IV	1.98		2.08		1.48	
	1.98		1.95		1.50	
		1.98		2.02		1.49
V	2.34		2.04		1.49	
	2.21		2.07		1.56	
		2.28		2.06		1.52
VI	2.27		2.23		1.74	
	2.30		2.39		1.76	
		2.28		2.31		1.75
VII	2.39		2.82		2.09	
	2.38		2.74		2.01	
		2.38		2.78		2.05
VIII	2.52		2.31		1.66	
	2.32		2.37		1.58	
		2.42		2.34		1.62
IX	2.62		2.78		2.13	
	2.54		2.78		2.09	
		2.58		2.78		2.11
X	2.90		3.16		2.55	
	2.95		3.13		2.44	
		2.92		3.14		2.50
XI	3.06		3.14		2.40	
	3.10		3.03		2.29	
		3.08		3.08		2.34
XII	4.67		4.55		3.80	
	4.79		4.49		3.66	
		4.73		4.52		3.73

[a] TAPPI Standard Method, T 223 m-48.

The extra distillate technique of Johansson, combined with ultraviolet absorption spectrophotometry has been used in this laboratory for several years in the estimation of the pentosans content of refined wood pulps.

EXPERIMENTAL

Preparation of Calibration Curve

A calibration curve was prepared by using xylose as the standard. Approximately 1 g. of ovendry xylose was dissolved in 13.2% HCl. The solution was diluted to 500 ml. with the same acid. Aliquots ranging from 5 to 90 ml. were transferred to distillation flasks which contained 20 g. of sodium chloride, 100 ml. of 13.2% HCl, a few glass beads, and a small piece of paraffin. Two hundred seventy-five milliliters of 13.2% HCl were introduced into the dropping funnel. The mixture was heated gently to boiling. Thereafter the distillate was collected in a 250-ml. volumetric flask at a rate of 2.5 ml. per min.; 25 ml. of acid was added to the contents of the distillation flask every 10 min. (The lower part of the stem of the funnel used in this laboratory comprises a 15-mm. glass bulb which is perforated in such a way that the flow of acid washes the walls of the flask.)

When 250 ml. of distillate had been collected, the flask was replaced by a graduated cylinder, and 20 to 25 ml. of distillate was collected and transferred to a small glass-stoppered flask. Ten milliliters of each of these two solutions were diluted to 1000 ml. with distilled water. With the extra distillate in a 10-mm. silica reference cell, the absorbence of the analytical solution was determined at 277 μ. A Beckman Model DU spectrophotometer was used for making the spectral absorbence measurements. Xylan equivalents (xylose × 0.88) were plotted against corresponding absorbence readings. The calibration curve was linear over the range covered by the use of 10 to 180 mg. of xylose.

Analysis of Pulp

The pulp samples listed in Table I were analyzed by the procedure described for the preparation of the calibration curve. Each sample consisted of approximately 2 g. of defibered pulp of known moisture content. Smaller samples may be used in the analysis of less highly purified pulps, or the two distillates resulting from each analysis may be further diluted in order to reduce the concentration of furfural to a practical working level. For comparison results are shown of the bromate–bromide analysis (TAPPI Standard Method T 223 m-48) and of the extra distillate modification of CCA 24. Each value reported in Table I represents the analysis of an individual sample. In no instance was a single distillate analyzed by two methods.

DISCUSSION AND CONCLUSIONS

The use of the extra-distillate technique obviates the necessity of applying an empirical correction for the presence of hydroxymethylfurfural; it renders the analysis less susceptible to the influence of variations in distillation conditions. Rate of distillation must, of course, be kept constant throughout any single determination, but the effect of variations that may be introduced from one analysis to the next is minimized by the use of distillate as the reference solution.

The application of colorimetry or of the extra-distillate modification of ultraviolet absorption spectrophotometry to the analysis of purified pulps eliminates much of the uncertainty that is involved in attempting to compensate for the presence of interfering materials by the use of an empirical correction factor. The extra-distillate modification of the ultraviolet spectrophotometric method offers the advantage of not requiring the carefully controlled conditions that are essential to the successful application of an unstable, multicomponent reagent to the development of color.

Ultraviolet absorption spectrophotometry is precise; its application to the determination of furfural in the presence of hydroxymethylfurfural is made relatively simple by the use of distillate in the reference cell; the analytical and reference solutions are noncorrosive.

LITERATURE CITED

1. Adams, G. A. and Castagne, A. E., *Can. J. Res.* 26B, 309–313 (1948).
2. Determination of Pentosans in Pulp, CCA 24. Swedish Association of Pulp and Paper Engineers. *Svensk Papperstidn.* 54, 179 (1951).
3. Browning, B. L., "The analysis of noncellulose polysaccharides," Chap. 30, in "Wood Chemistry," by Wise, L. E. and Jahn, E. C., Vol. 2, New York, Reinhold, 1952.
4. Wilson, W. K. and Mandel, J., *Tappi* 43, No. 12: 998–1006 (1960).
5. Launer, H. F. and Wilson, W. K., *J. Research Natl. Bur. Standards* 22, 471–481 (1939).
6. Johansson, Axel, *Svensk Papperstidn.* 55, 820–828 (1952).
7. Smith, E. D. and Rogers, L. N., *Anal. Chem.* 25, 931–933 (1953).
8. Smith, E. D. and Rogers, L. N., *Tappi* 36, No. 9: 390–392 (1953).
9. Stillings, R. A. and Browning, B. L., *Ind. Eng. Chem. Anal. Ed.*, 12, 499–502 (1940).

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Analysis of Stone Cells from Bigtooth and Quaking Aspens

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Although stone cells are an abundant structural element in the bark of many trees, very little is known about their composition. A quantity of stone cells sufficient for approximate chemical analyses was obtained directly from methanol-extracted, air-dried, inner bark of bigtooth aspen by simply grinding and screening out the larger, more resistant particles. In the case of quaking aspen, the grinding of the inner bark had to be supplemented by sedimentation and, to some extent, by picking out contaminating particles. The analyses showed that the stone cells of both bigtooth and quaking aspen barks are similar to the wood of these species. Since they were relatively free from the resins, tannins, proteins, and other plant constituents which make the study of bark lignins difficult, they appear to be good starting materials for further studies.

This Work with stone cells came about through the observation that a portion of the inner bark of a bigtooth aspen tree was unusually resistant to grinding in the Wiley mill. When the coarse particles were separated, it was noted that they consisted almost entirely of stone cells (Fig. 1). Stone cells are a subgroup of a type of plant cell known as sclerids, that is, cells which have developed thick, lignified, secondary walls. In particular, stone cells are relatively short, roughly cubic in shape, and often appear to be solid. They frequently occur in clusters which are rather firmly cemented together; the hard, gritty granules in the flesh of some pears are a familiar example of stone cells.

Stone cells and other forms of sclerids are abundant in the barks of many pulpwood species. In spite of this, very little appears to be known about their composition (*1*). An analysis of pear stone cells indicated that they "are a lignified cellulose resembling in composition the forest woods rather than annual lignocelluloses such as jute" (*2*). Accordingly, the availability of a sample from bigtooth aspen bark of stone cells presented an opportunity to compare the composition of these characteristic bark elements with that of bark and wood.

SEPARATION OF STONE CELLS

Bigtooth Aspen, Populus grandidentata

The stone cells from the inner bark of bigtooth aspen, which had previously been extracted with methanol and air-dried, were obtained directly by sieving out the coarser particles after chips of the bark had been passed through a Wiley mill.

Quaking Aspen, Populus tremuloides

A sample of methanol-extracted, airdry bark chips was ground once through a 0.312-in. diameter screen in the Wiley mill. The coarsely ground meal was screened on no. 20, 30, and 40 sieves by mechanical shaking. Microscopic examination showed that the 30–40 mesh fraction contained the most stone cells, but crystalliferous strands and phloem parenchyma also were present. Screening did not give any further separation.

Test tube sedimentation experiments showed that a 1:1 volume ratio of carbon tetrachloride and ethylene dichloride gave a lighter and a heavier fraction. This ratio of the liquids was tried on a larger scale.

Thirty-five grams of the 40-mesh fraction was weighed out and mixed with 400 ml. of liquid. After sedimenting overnight, the floating fraction was skimmed off and the heavier fraction suction-filtered. Both fractions were air-dried and then examined microscopically. The floating fraction contained equal amounts of stone cells and phloem parenchyma, and very little of the crystalliferous strands. The sediment contained about 70% crystalliferous strands, the remainder being stone cells and parenchyma.

This procedure was repeated with the remainder of the 40-mesh fraction, weighing 39 g. The two floating fractions were combined, as were the sediments.

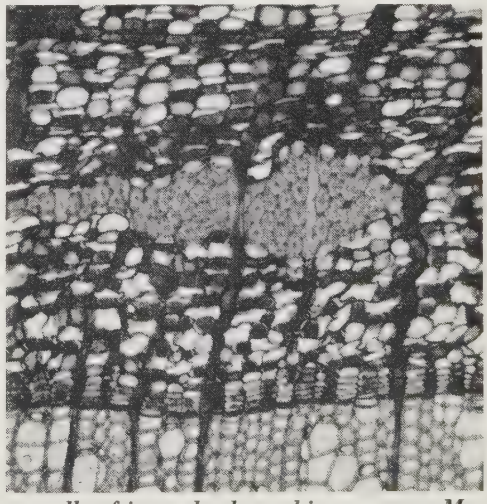

Fig. 1. Stone cells of inner bark quaking aspen. Magnification 100×

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The floating fraction, in which the stone cells were concentrated, was too contaminated for analysis. The stone cells were placed on a no. 40 screen and by gentle agitation and blowing some of the contaminants were removed; but this was not a very satisfactory way to purify the stone cells.

By again trying different ratios of carbon tetrachloride and ethylene dichloride it was found that in a 1:3 mixture of carbon tetrachloride-to-ethylene dichloride the parenchyma floated and the "stone cells" sedimented. It was noticed that there were two layers in the sediment. The bottom one consisted of bright yellow, massive particles, and the top layer of brownish linear particles. By carefully decanting the solvent, after the floating layer was removed, the exposed top layer of the sediment could be scraped off with a spatula. This layer was crystalliferous strands. The bottom layer of stone cells was almost free from contamination.

The 30-mesh fraction was processed in the same manner, with one step being added; i.e., a large part of the long linear particles were removed with tweezers.

A portion of the 20-mesh fraction was reground through the 20-mesh screen in the micro-Wiley mill. This material was screened on a no. 40 sieve and the sample sedimented as in the preceding fractions. The stone cell fraction was further purified by removing the linear particles with tweezers.

All the purified stone cell fractions from quaking aspen, a total of 20.8 g., was combined for analysis.

RESULTS

Table I shows the results of the analyses. In work of this kind, little more can be said than what the figures themselves show. In general, there is considerable similarity between the composition of the stone cells and that of the wood. This result supplements the finding that the stone cells from pears are a woody lignocellulose. Specifically, the values for hot-water extract, nitrogen, and uronic anhydride are close to those for wood. The value for the degree of polymerization, estimated from the purified cellulose nitrates, is high in relation to the usual values of pulps, but is consistent with the values estimated for wood cellulose prior to pulping. Of the other assays, the ash and the xylan appear to be somewhat higher than the figures for the wood; the acetyl, holocellulose, and α-cellulose appear to be somewhat lower. Perhaps the greatest difference is in the low value for glucose, expressed as glucosan, in comparison with that expected from the α-cellulose content. However, there is an appreciable deficit in the glucose recovered after hydrolysis from both wood and stone cells, although the deficit for the stone cells is larger. The apparent density of about 1:1 is consistent with the microscopic appearance of stone cells, which show very little free space or lumen inside the greatly thickened secondary wall. The true density of approximately 1.41 g./cc. approaches the figure 1.56 g./cc. assigned as the density of wood substance.

Table I. Composition of Stone Cells, Inner Bark, and Wood in Aspen

Analyses	Bigtooth aspen			Quaking aspen		Method (4)
	Stone cells,[a] %	Inner bark,[c] %	Wood,[c] %	Stone cells,[a] %	Wood, %	
Moisture	5.6[b]	7.0	5.8	6.7[b]		T 210 m-58
Alcohol–benzene extract	2.6		2.4[d]	4.6		T 6 m-59
Hot-water extract	2.6	6.0	2.4	3.3	2.7	T 1 m-59
Ash	2.6[b]	4.4	0.3	2.5[b]	0.4	T 15 m-58
$CaC_2O_4 . H_2O$	+			+		X-ray diffraction
Nitrogen	0.1	0.3	0.1	0.4	0.1	T 418 m-50
Uronic anhydride	5.1[b]	9.0	4.9	5.2	5.1	(5)
Acetyl	4.2		5.5[d]	4.0	5.1[d]	(6)
Lignin	23.9	22.7	18.2	23.2[b]	16.9	T 13 m-54[g]
Methoxyl in lignin	18.9			18.0[b]		T 2 m-43
Holocellulose	66.1[b]		78.0[de]	72.7[b]	79.5[df]	(7)
Sugars, after hydrolysis						(8)
Dextran	32.3		45.0	27.2	45.8	
Xylan	20.7		18.9	22.2	16.0	
Galactan	1.0		1.1	1.2	1.2	
Mannan	0.7		2.4	0.4	2.1	
Araban	1.6		0.7	1.9	0.7	
Alpha-cellulose	43.0[b]			45.2[b]	51.0[d]	T 429 m-48 (grav.)
Nitrocellulose, D.P.	3996			3872		(4)
True density, g./cc.	1.417			1.408		(4)
Apparent density, g./cc.	1.106[b]			1.098[b]		

[a] Assays were made with material <60 mesh, except as noted.
[b] Assays were made with 40–60 mesh material.
[c] I.P.C. data, unpublished.
[d] Ritter, G. A. and Hossfeld, R. L., Lake States Aspen Report No. 18, U.S.L.S. Forest Expt. Sta., 1947.
[e] I.P.C. data, unpublished.
[f] I.P.C. data, unpublished.
[g] Five hours primary hydrolysis.

LITERATURE CITED

1. E. G., no reference to stone cells or to sclerids appears in the indices to: Wise, L. E., and Jahn, E. C., "Wood Chemistry," 2nd ed., New York, Reinhold, 1952.
2. Doreé, C. and Barton-Wright, E. C., *Biochem. J.* 20: 502 (1926).
3. Schorger, A. W., "The Chemistry of Cellulose and Wood," p. 19. New York, McGraw-Hill, 1926.
4. Complete descriptions of the methods used for Nitrocellulose Viscosity (D.P.), Apparent Density, and True Density are on file at The Institute of Paper Chemistry, Appleton, Wis.
5. Browning, B. L., *Tappi* 32: 119 (1949).
6. Whistler, R. L. and Jeanes, A., *Ind. Eng. Chem., Anal. Ed.* 15: 317–318 (1943).
7. Wise, L. E., Murphy, M., and D'Addieco, A., *Paper Trade J.* 122: 35–43 (1946).
8. Piper, C. V. and Bernardin, L. J., *Tappi* 41: 16 (1958).

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Stabilization Studies on Cotton and Wood Cellulose Nitrates

PAUL E. GAGNON, BRIAN T. NEWBOLD, and J. THOMAS

Unstabilized cotton and wood cellulose nitrates were heated in water at different temperatures for various lengths of time, and also boiled in water for different periods of time, up to 1 hr. The sulfate content of the cellulose nitrates decreased steadily as the water temperature increased. By means of the boiling treatment the sulfate contents decreased considerably during the first 15 min. and then remained approximately constant. It was found that for unstabilized cotton and wood cellulose nitrates about 76% of the sulfate content could be removed by either heating in water at 69°C. for 24 hr. or boiling in water for 1 hr. No decomposition of the nitrate ester occurred, there being no significant change in the nitrogen contents. Cotton, and wood cellulose nitrates, which had been boiled in water for 1 hr. were stored at about 5°C. for 135 days. These products were found to have remained chemically stable over that period. There was no significant decrease in the nitrogen contents or increase in the acidities.

Many papers have been published on the stabilization of cellulose nitrate. It was pointed out in early work on the subject that a good stabilization method must be rapid and be accompanied by only a little degradation of the cellulose nitrate and small decrease in nitrogen content (*1*). Various methods have been employed for the stabilization of cellulose nitrate. One step in the commercial stabilization, for example, involves milling or pulping, and it is said that such treatment washes out residual acid from the fibers (*2*). Cellulose nitrate that has been made commercially contains a small amount of sulfate that is not removed by the stabilization procedures. Kullgren (*3*) stated that cellulose nitrate has a sulfate content of about 0.2%, expressed as sulfuric acid, part of which is an active ester and binds cations. The activity was soon lost on boiling in water or 0.05*N* HNO_3, and the boiled product on washing for many hours became almost acid free.

Kullgren (*4*) also mentioned that boiling with water was used technically for producing stable cellulose nitrate. The sulfate ester in unstabilized cellulose nitrate can be removed by prolonged boiling in water maintained just alkaline, the decomposition being most rapid when the boiling is carried out under slightly acid conditions (*5*). Chédin and Tribot (*6*) found that when cellulose nitrate was boiled with water for an extended period of time, almost all the residual sulfuric acid was taken off in the first 90 hr. Recently, Bricknell, *et al* (*7*), studied the sulfate content of stabilized cellulose nitrate and found that it could almost all be removed by boiling in water for 48 hr. Another method that has often been used in the laboratory to remove most of the sulfate present in crude cellulose nitrate is treatment with a 50% solution of ethanol, which swells the fibers and releases free acid (*8*).

The main object of the present work was to study the removal of sulfate from both unstabilized cotton and wood cellulose nitrates (nitrogen content range from 11 to 12%), by means of (1) extended heating in water, and (2) boiling in water for short periods of time, with a view to avoiding degradation of the cellulose nitrates and preserving the nitrogen content.

EXPERIMENTAL

Materials

High-grade cotton linters were supplied by the Hercules Powder Co. and wood cellulose (*9*) by Canadian Industries Ltd. Mixed acids containing sulfuric and nitric acids were prepared and analyzed by the standard method.

Cellulose Nitrates

Cotton linters and wood cellulose were nitrated with a series of mixed acids, by the method given previously (*8, 10*). The nitrations were performed under similar conditions. Typical experimental conditions for nitrations of wood cellulose were given in a previous publication (*11*). The nitrogen contents of the unstabilized cellulose nitrates were determined by the Devarda method and the total sulfate contents were obtained by the barium sulfate method (*9*).

The unstabilized cotton and wood cellulose nitrates prepared in the present work had nitrogen contents varying from 11.3 to 12.1%, and 10.9 to 11.9%, respectively. The total sulfate contents varied from 0.63 to 0.90%, and 0.93 to 1.58%, expressed as per cent sulfuric acid.

Heat Treatments of Cellulose Nitrates

Well-washed, 20-g. samples of unstabilized cotton and wood cellulose nitrates were placed in 3-liter flasks and 1 liter of distilled water added. The material was then heated, with occasional shaking, for various periods of time 0.5–24 hr.) at different temperatures (34–69°C.) in a constant temperature bath. After the heating was completed, the cellulose nitrate samples were removed by filtration and then allowed to soak in 1 liter of water, with occasional stirring for 10 min. The samples were filtered off and again soaked in 1 liter of water for 5 min., with occasional stirring. This washing procedure was repeated four more times, the products were then dried by centrifuging (*10*) and samples were set aside for moisture, nitrogen, and sulfate determinations.

PAUL E. GAGNON, present address: International Atomic Energy Agency, Vienna 1, Austria. BRIAN T. NEWBOLD, Professor of chemistry, Chemistry Department, St. Joseph's University, Moncton, N. B., Canada. J. THOMAS, Research Assistant, Chemistry Department, Laval University, Quebec, Que., Canada.

Contribution from the Chemistry Department, Laval University, Quebec, Que., with financial assistance from the Defense Research Board of Canada.

Table I. Changes in the Nitrogen and Sulfate Contents of Cellulose Nitrates Caused by Heating in Water

Expt. no.	Temp., °C.	Time, hr.	Cotton cellulose nitrate			Wood cellulose nitrate		
			N, %	Sulfate, % H_2SO_4	% of original sulfate	N, %	Sulfate, % H_2SO_4	% of original sulfate
1	25	0	11.3	0.85	100	10.9	1.34	100
	34	24	11.3	0.57	66	10.8	1.20	81
2	25	0	11.4	0.83	100	11.0	1.58	100
	42	24	11.5	0.69	79	11.1	0.87	59
3	25	0	11.4	0.90	100	11.0	1.52	100
	55	24	11.3	0.39	45	11.0	0.71	48
	65	24	11.3	0.25	29	11.0	0.52	35
4	25	0	11.3	0.88	100	10.9	1.49	100
	69	24	11.4	0.21	24	10.9	0.35	24

In other experiments, unstabilized cotton and wood cellulose nitrates were treated with boiling water. Well-washed 20-g. samples were placed in 3-liter flasks along with 1 liter of distilled water and the contents were boiled for various lengths of time (15–60 min.), with occasional stirring. After this treatment, the cellulose nitrates were well washed, as described above, and portions set aside for analysis.

Storage of Cellulose Nitrates

Moist samples of unstabilized cotton (11.5% N) and wood (11.0%N) cellulose nitrates were stored in sealed flasks at about 5°C., for 135 days. A sample of each of these cellulose nitrates was boiled in distilled water for 1 hr., the resulting product was well washed and stored in the moist condition at about 5°C., also for a period of 135 days. The nitrogen contents of the heat-treated cellulose nitrates were determined before and after storage, and the acidities of both the unstabilized and the heat-treated cellulose nitrates were also determined before and after storage. The latter values were obtained by titration in 90% acetone solution with 0.01*N* KOH, using a Macbeth pH meter (*11*). The acidities were expressed as milliliters of 0.01*N* KOH per gram of cellulose nitrate.

The nitrogen contents of the unstabilized cotton and wood cellulose nitrates after storage were 11.4% and 11.0%, respectively, and the acidities before and after storage were 8.81, 14.42 ml./g., and 8.02, 16.37 ml./g., respectively.

The nitrogen contents of the heat-treated cotton and wood cellulose nitrates before and after storage were about the same, namely, 11.5% and 11.0%, in each case. The corresponding acidities were 2.44, 2.95 ml./g, and 2.21, 2.86 ml./g.

DISCUSSION

Heating-in-Water Treatment of Cellulose Nitrates

The sulfate content of well-washed cellulose nitrate which has been prepared from mixed acid varies from about 0.2 to 3.0%, expressed as sulfuric acid, depending upon the composition of the nitric acid–sulfuric acid mixture. The total sulfate content tends to increase with decreasing nitrogen content (*8*, *12*, *13*).

In the present work, one of the main aims was to reduce the sulfate contents of unstabilized cellulose nitrates by some mild means which would not cause decomposition of the nitrate ester. Accordingly, both cotton and wood cellulose nitrates were heated at different temperatures in the presence of water, for varying lengths of time. It was found that heating for a short time at temperature less than 50°C. did not affect the sulfate contents greatly. For instance, for an unstabilized wood cellulose nitrate containing 11.0% nitrogen there was only a decrease of 5% in the total sulfate content when it was heated at 50°C. for 30 min. A similar result was obtained when the heating period was extended to 3 hr. However, when a cotton cellulose nitrate containing 11.7% nitrogen was subjected to the latter treatment, the total sulfate content decreased by some 25%.

In view of the above results, it appeared worthwhile to extend the period of heating considerably and to study the effect of increasing water temperature on the total sulfate content. In a series of experiments, cotton cellulose nitrates containing about 11.3% nitrogen, and wood cellulose nitrates containing about 11.0% nitrogen, were heated for 24 hr. at various temperatures. The results are given in Table I. In both types of cellulose nitrate the sulfate content decreased steadily with increasing water temperature and, furthermore, the nitrate ester remained stable under this treatment, even at 69°C. There was no significant decrease in the nitrogen content. The small differences between nitrogen contents were within the

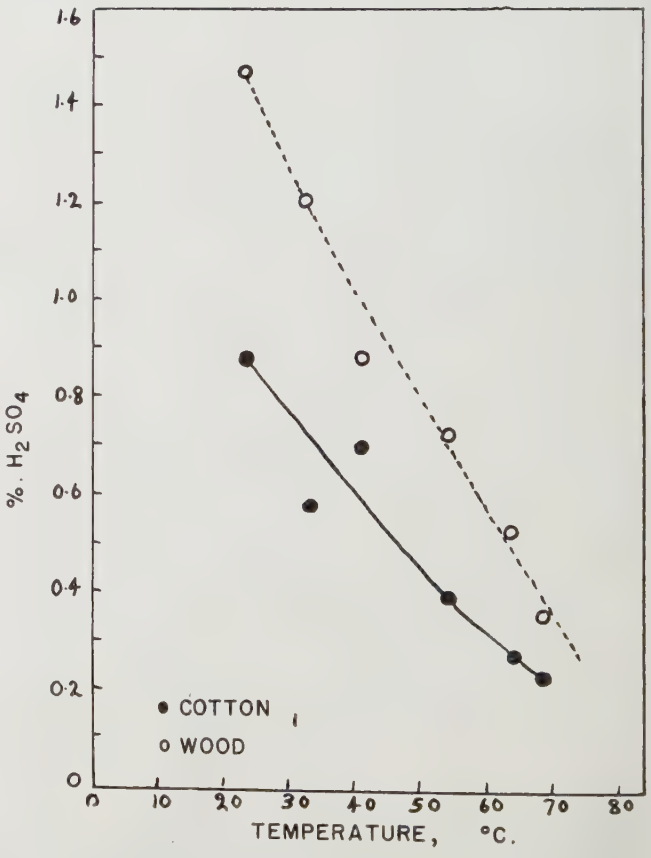

Fig. 1. Relation between sulfate content of cellulose nitrates and water temperature (period of heating = 24 hr.)

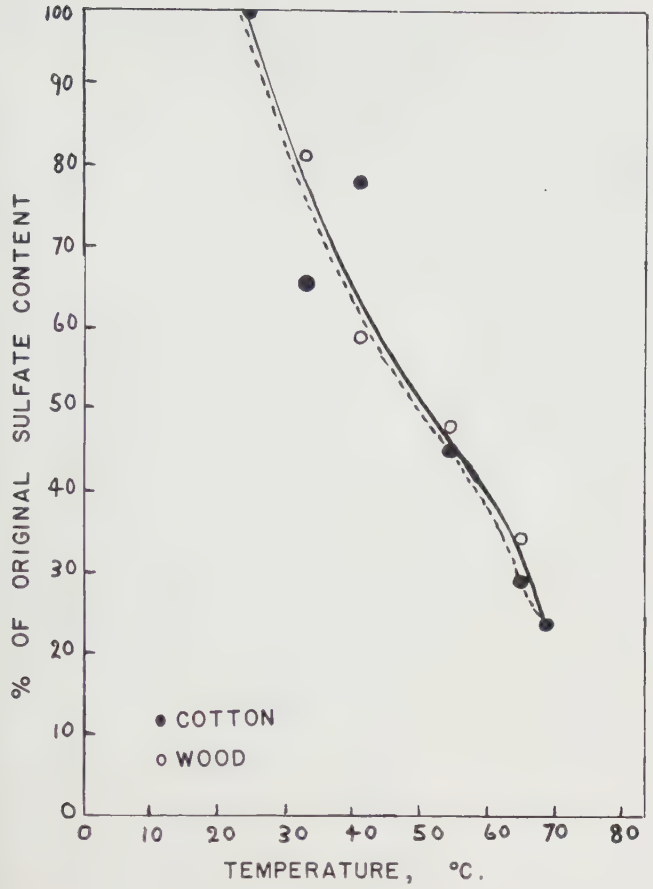

Fig. 2. Percentage decrease in sulfate content of cellulose nitrates with increasing water temperature (period of heating = 24 hr.)

experimental error of the method used to determine nitrogen. It has been shown that the nitrate ester of unstabilized cellulose nitrate does not decompose when the latter is hydrolyzed in acetone solution at a temperature as high as 45°C. (*14*).

Figure 1 shows the relation between sulfate content and water temperature. The two points on the curves corresponding to a temperature of 25°C., represent the average sulfate contents of the cellulose nitrates before treatment and are therefore used as reference points. The initial sulfate content of the wood cellulose nitrate is higher than that of the cotton cellulose nitrate, which agrees with our previous findings regarding the sulfate content of cotton and wood cellulose nitrates prepared under similar conditions (*15*). Furthermore, although the sulfate contents decrease steadily with increasing water temperature, the sulfate content of the wood cellulose nitrate always remains higher than that of the cotton cellulose nitrate, when both have received the same treatment, (Fig. 1). However, by comparing the sulfate contents of the two cellulose nitrates after treatment, with the original sulfate contents (Fig. 2), it is seen that the effect of heating in water is about the same for both types. The two curves obtained are very similar. The point corresponding to a temperature of 25°C., for both curves, represents the original sulfate contents as 100%. Figure 2 shows that the maximum loss of sulfate in both types of cellulose nitrate is 76%, which occured when the samples were heated in water at 69°C. for 24 hr.

Short-Boiling Treatment of Cellulose Nitrates

Various methods have been employed for the stabilization of cellulose nitrate, such as refluxing in water for 30 hr., and boiling in distilled water for 9 hr. with the water being changed every 3 hr. (*7*). When an attempt is made to remove all the sulfate from cellulose nitrate, considerable degradation of the nitrate ester occurs, with consequent decrease in the nitrogen content. For instance, Bricknell, *et al.* (*7*), tried to remove the sulfate from a stabilized cotton cellulose nitrate containing 0.06% sulfate, by digesting in water with vigorous stirring at a temperature of 90 to 95°C. for 48 hr., the water being changed after 24 hr. This treatment removed 87% of the original sulfate present, but degradation of the cellulose nitrate occured.

In this work, the effect of boiling in water for short periods of time on the sulfate and nitrogen contents of both unstabilized cotton and wood cellulose nitrates was also studied. Since over three-quarters of the sulfate content had been removed by heating in water at 69°C. for 24 hr. it was felt that boiling in water for a much shorter time might give even better results. Accordingly, unstabilized cotton and wood cellulose nitrates were boiled in distilled water for various lengths of time, up to 1 hr. The results are given in Table II.

After only 15 min. the sulfate contents of both types of cellulose nitrate had decreased considerably, but extending the treatment to 1 hr. did not cause any further great loss of sulfate for the cotton cellulose nitrate. There was a slight decrease in the sulfate content of the wood cellulose nitrate. The nitrate ester remained stable in both cases, as shown by the constant values of the nitrogen contents. The reproducibility of the results is shown in experiment 2 and 3 (Table II), where the cellulose nitrates were boiled in water for 1 hr. The relation between the sulfate content and the boiling time is shown in Fig. 3. It is seen that after the great decrease in sulfate content caused by 15-min. boiling the remaining sulfate is much more difficult to remove. The sulfate content of the wood cellulose nitrate was always higher than that of the cotton cellulose nitrate. For instance, after 1-hr. boiling the sulfate contents of the wood and cotton cellulose nitrates were 0.30% and 0.20%, respectively.

Table II. Changes in the Nitrogen and Sulfate Contents of Cellulose Nitrates Caused by Boiling in Water

Expt. no.	*Time, min.*	*Cotton cellulose nitrate*			*Wood cellulose nitrate*		
		N,%	*Sulfate, % H_2SO_4*	*% of original sulfate*	*N, %*	*Sulfate, % H_2SO_4*	*% of original sulfate*
1	0	11.6	0.74	100	11.0	1.50	100
	15	11.5	0.21	28	11.0	0.37	28
2	0	11.6	0.76	100	10.9	1.11	100
	30	11.5	0.20	27	10.9	..	..
	60	11.6	0.16	21	11.0	0.27	20
3	0	11.5	0.76	100	11.0	1.41	100
	60	11.5	0.20	27	11.0	0.30	22
4	0	12.1	0.63	100	11.9	0.93	100
	60	—	0.29	46	—	0.52	56

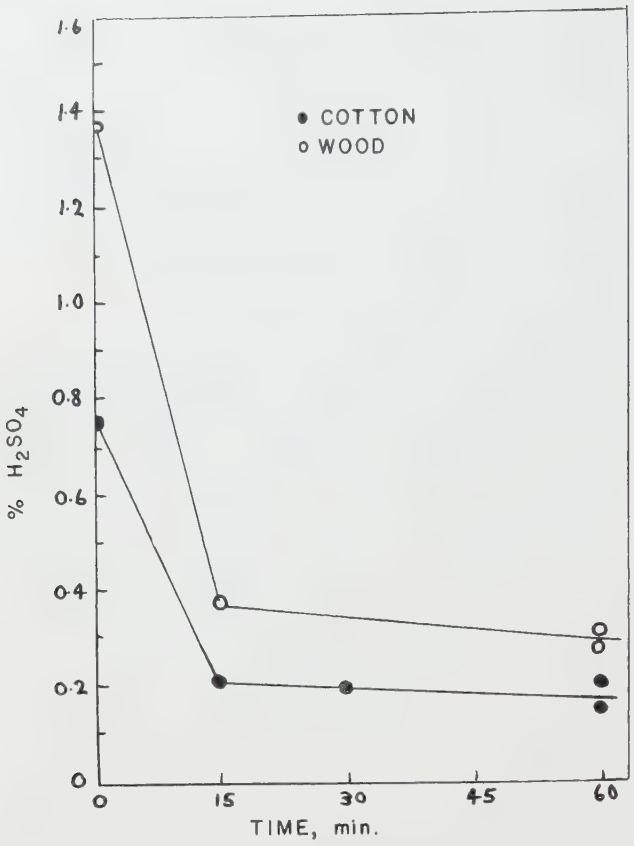

Fig. 3. Relation between sulfate content of cellulose nitrates and boiling time

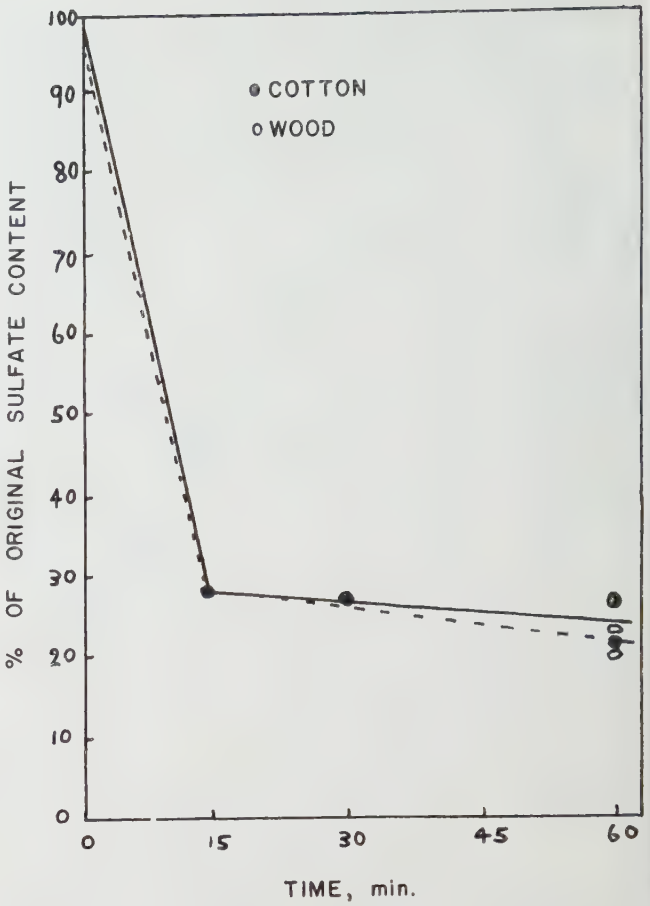

Fig. 4. Percentage decrease in sulfate content of cellulose nitrates with increasing boiling time

The effect of boiling, expressed as percentage decrease in sulfate content, was about the same for both types of cellulose nitrate, as shown in Fig. 4. For example, the loss of sulfate after 15-min. boiling was 72% in both cases, and the loss of sulfate after 1-hr. boiling was 76% and 79%, respectively, for the cotton and wood cellulose nitrate.

Other cotton and wood cellulose nitrates having higher nitrogen contents were also boiled in water. Some results are shown in experiment 4 (Table II). One hour of boiling caused only 54% and 44% decreases in sulfate content of the cellulose nitrates. It was more difficult to remove sulfate from cellulose nitrates having relatively low sulfate contents, as expected.

Comparison of the two heat treatment methods reveals that a 1-hr. boiling treatment was equivalent to heating in water at 69°C. for 24 hr., with regard to loss of sulfate for both types of unstabilized cellulose nitrate (Figs. 2 and 4). For instance, for cotton cellulose nitrate the sulfate contents after these two treatments were in close agreement, being 0.21% and 0.20%, respectively. (Figs. 1 and 3).

Storage of Cellulose Nitrates

It is well known that cellulose nitrates gradually deteriorate in storage, and that there is an accompanying decrease in the nitrogen content. In the present work, the effect of storage upon the chemical stability of heat treated cellulose nitrates was investigated. Cotton and wood unstabilized cellulose nitrates (11.5% and 11.0% nitrogen, respectively), were boiled in water for 1 hr., well washed, and then stored in moist condition at about 5°C. for 135 days. Samples of unstabilized cellulose nitrates were also stored at 5°C. for the same period of time. There was no significant decrease in the nitrogen content of any of the samples. However, in the case of the unstabilized wood cellulose nitrate there was a definite increase in acidity. In contrast, there was no significant increase in acidity in the heat-treated sample. Similar results were obtained with the cotton cellulose nitrates.

LITERATURE CITED

1. Robertson, R., *J. Soc. Chem. Ind.* 25: 624 (1906).
2. Davis, T., "Chemistry of Powder and Explosives," New York, John Wiley & Sons, 1943.
3. Kullgren, C., *Svensk Kem. Tidskr.* 53: 233 (1941).
4. Kullgren, C., *Svensk Kem. Tidskr.* 56: 221 (1944).
5. Doree, C., "Methods of Cellulose Chemistry," London, Chapman and Hall, 1950.
6. Chédin, J. and Tribot, A., *Mém. Services chim. état (Paris)* 32: 157 (1945).
7. Bricknell, A. G., Trevoy, L. W., and Spinks, J. W. T., *Can. J. Chem.* 35: 704 (1957).
8. Gagnon, P. E., Keirstead, K. F., and Yamasaki, D., *Can. J. Tech.* 34: 477 (1957).
9. Keirstead, K. F. and Myers, J. L., *Can. J. Chem.* 32: 566 (1954).
10. Gagnon, P. E., Keirstead, K. F., Newbold, B. T., and Thomas, J., *Can. J. Chem.* 36: 1039 (1958).
11. Gagnon, P. E., Keirstead, K. F., and Newbold, B. T., *Can. J. Chem.* 36: 291 (1958).
12. Miles, F. D., "Cellulose Nitrate," Toronto, Clarke, Irwin & Co., 1955.
13. Gagnon, P. E., Keirstead, K. F., and Newbold, B. T., *Can. J. Chem.* 36: 212 (1958).
14. Gagnon, P. E., Keirstead, K. F., Newbold, B. T., and Thomas, J., *Tappi* 41, No. 9: 515 (1958).
15. Gagnon, P. E., Keirstead, K. F., and Newbold, B. T., *Can. J. Chem.* 36: 215 (1958).

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ASSOCIATION NEWS AND EVENTS

National Conferences

1961

Sixth Deinking Conference, Oct. 4–6, 1961, Hotel Harris, Kalamazoo, Mich.

Sixteenth Plastics-Paper Conference, Oct. 9–11, 1961, French Lick Sheraton Hotel, French Lick, Ind.

Sixteenth Engineering Conference, Oct. 15–19, 1961, Shoreham, Hotel, Washington, D. C.

Fifteenth Alkaline Pulping Conference, Nov. 1–3, 1961, Rice Hotel, Houston, Tex.

1962

Forty-seventh Annual Meeting, Feb. 18–22, 1961, Commodore Hotel, New York, N. Y.

Thirteenth Coating Conference, May 14–16, 1962, Netherland-Hilton Hotel, Cincinnati, Ohio.

Thirteenth Testing Conference, Sept. 18–20, 1962, Benjamin Franklin Hotel, Philadelphia, Pa.

Seventeenth Plastics-Paper Conference, Oct. 8–11, 1962, Statler-Hilton Hotel, Boston, Mass.

Seventeenth Engineering Conference, Oct. 14–19, 1962, Queen Elizabeth Hotel, Montreal, Que.

Twelfth Corrugated Containers Conference, Oct. 23–26, 1962, Statler-Hilton Hotel, Boston, Mass.

Sixteenth Alkaline Pulping Conference, Oct. 31, Nov. 1–2, 1962, DeSoto Hotel, Savannah, Ga.

1963

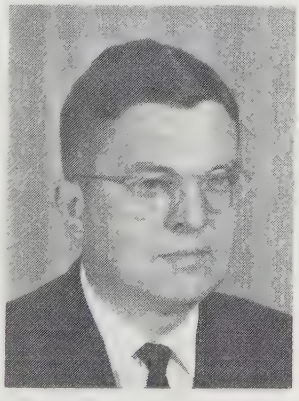
Fourteenth Coating Conference, May 19–22, 1963, Concord Hotel, Kiamesha Lake, N. Y.

Thirteenth Corrugated Containers Conference, Oct. 22–25, 1963, Statler-Hilton Hotel, Detroit, Mich.

Eighteenth Engineering Conference, Oct. 28–31, 1963, Netherland-Hilton Hotel, Cincinnati, Ohio.

Testing Division

Herbert A. Smith, technical director of The Mead Corp.'s Chillicothe Division, has been appointed general chairman of the Testing Division of the Technical Association of the Pulp and Paper Industry by President Harold M Annis of Oxford Paper Co.



Herbert A. Smith, The Mead Corp.; general chairman, Testing Division

Mr. Smith received a B.A. in chemistry at Ohio Wesleyan in 1937 and was a special student at The Institute of Paper Chemistry in Appleton, Wis. He has been active in TAPPI Testing Division affairs for many years and has contributed directly to the development of many methods for the evaluation of pulp and paper products. He is a past chairman of the Ohio Section of TAPPI, and a member of the Education Committee. He is a member of the American Chemical Society and the Ohio Academy of Science.

Southern California TAPPI

TAPPI members in Southern California held their first organizational meeting at the Rodger Young Auditorium in Los Angeles on August 8. A total of 84 men from 11 paper companies and a number of supplier organizations attended the dinner meeting, which featured a discussion of "Proposed Legislation Affecting the Paper Industry" by T. Knight of the California Manufacturers Association.

At a business meeting preceding the dinner the group voted to request status as the Southern California District of the Golden Gate Section. Articles of Organization to govern the operations of the proposed district were approved, and the following officers were elected:

Chairman: E. Tonjes, The Flintkote Co., Los Angeles
Vice-Chairman: T. Pringle, Jr., Menasha Container of California, Anaheim
Secretary: D. T. Snell, Continental Can Co., Inc., Los Angeles
Treasurer: L. R. Giersch, Crown Zellerbach Corp., Los Angeles

S. Feldman, The Flintkote Co., Los Angeles, was appointed chairman of the Membership Committee, and L. L. Larson, Potlatch Forests, Inc., Pomona, was named chairman of the Publicity Committee.

Meetings will be held at the Rodger Young Auditorium on the second Tuesday of even-numbered months. The technical program will begin at 4 p.m., to be followed by refreshments at 5:30 p.m. and the dinner at 6 p.m. Notices of these meetings will be mailed to TAPPI members in the area and to all others who have expressed their interest in the activities of this group. The next meeting will be held October 10.

Maine–New Hampshire

The Maine-New Hampshire Section will hold its fall meeting at the Mountain View House, Whitefield, N. H., on Oct. 20–21, 1961.

The technical program which has been arranged by Paul Goodloe of the Brown Co. will be devoted to high-speed, high temperature continuous cooking of hardwoods and softwoods. Both kraft and sulfite cooking will be considered. All of the papers will be presented by members of the staff of the Pandia Div. of The Black-Clawson Co.

On Saturday morning, October 21 a visit will be made to the mill of the Brown Co. at Berlin, N. H. to view the pilot plant continuous cooking unit in operation. A member of the staff of the Pulp and Paper Research Institute of Canada will be present at the Friday meeting to discuss the process.

Room reservations should be made directly with the Mountain View House in Whitefield, N. H.

1961 Lignin Symposium

The 1961 Lignin Symposium was held August 14–16, 1961 at the Edgewater Beach Hotel in Chicago, Ill. This symposium was a renewal of other lignin roundtables sponsored by TAPPI in previous years. There were 144 people in attendance with 12 countries represented besides the United States. J. R. Salvesen, Marathon Div., American Can Co., Rothschild, Wis., was general chairman of the Symposium. He is also chairman of the TAPPI Wood Chemistry Committee, the sponsoring committee of the symposium. W. M. Hearon, Crown Zellerback Corp., 1 Bush St., San Francisco, Calif., arranged the outstanding technical program. Unfortunately, Dr. Hearon was unable to attend the symposium because of illness.

Authors and International Visitors Reception

O. T. Moldenhauer, Anders Bjorkman, J. Richter Salvesen, H. F. Lewis, F. E. Brauns, and J. M. Pepper

T. Enkvist, O. Ellefsen, Waldemar Jensen, J. Richter Salvesen, Lennart G. Stockman, R. G. Macdonald, and J. Gierer

J. Gierer, David Hogan, Mrs. Holger Erdtman, Joseph Marton, Holger Erdtman, and T. Enkvist

F. J. Ball, J. Giguere, David Craig, Motoyoshi Oshima, W. H. Gloz, and D. L. Brink

Karl Keirstead, W. A. Kraske, J. W. T. Merewether, E. E. Harris, and Kyosti Sarkanen

Lennart G. Stockman, S. Hossain, J. W. T. Merewether, Mrs. A. J. Wiley, J. Benko, Mrs. J. Benko, and A. J. Wiley

R. F. Benning, J. S. Barton, J. J. Lothe, M. A. Baum, and A. S. Gregory

Anders Bjorkman, R. H. Farmer, Kan. Kashima, F. E. Brauns, and Haruro Tabata

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A reception was held Sunday evening for the authors and international visitors, the latter group comprising people from Australia, Brazil, Canada, Denmark, England, Finland, France, Germany, Italy, Japan, Norway, and Sweden.

J. R. Salvesen opened the symposium at 9:00 a.m. on Monday with the following remarks:

Introduction to TAPPI 1961 Lignin Symposium—J. Richter Salvesen

On behalf of the TAPPI Wood Chemistry Committee, I have the pleasure to welcome you to a three-day symposium pertaining to utilization of lignin and other by-products resulting from the wood pulping processes.

We have organized this symposium around the theme: "Lignin—Greater Utilization Through Better Understanding." A more complete, economically sound utilization of the major wood components is today of importance all over the world. The good attendance here, with international representation among the speakers and on the registration list, demonstrates the significance of the topics we are here to discuss.

We are glad to have with us English speaking representatives from such far away places as England and Australia. To the participants from the Scandinavian countries I am addressing a few words in their native tongue: Velkommen til disse möte-dager for utveksting av kundskaper av felles interesse og for at stifte og utvide venskapelig kontakt for fremtiden. I want to welcome the German speaking participants—Ihr Besuch macht uns Freude, wir schätzen Ihre Anwesenheit und Mitarbeit und hoffen dass Sie unsere Sitzungen inhaltsreich und anregend finden werden. We are also pleased to have participants from Japan and I regret that my linguistic limitation prevents me from also greeting them in their own language.

In a sense this is the fourth symposium TAPPI has sponsored in this country for stimulating interest in and exchange of knowledge of the noncellulose components of wood. The first such symposium was held in Appleton, Wis., in 1947, organized by Dr. Harry F. Lewis, vice-president of The Institute of Paper Chemistry. Three years later, in 1950, a second such meeting was held at The Institute of Paper Chemistry in Appleton and known as a Lignin Round Table, again under the energetic chairmanship of Dr. Lewis. And then in 1956, a symposium of this type was held at the Paper Institute, this time devoted primarily to the biogenesis of lignin. These previous lignin symposiums were mainly academic in nature. The papers presented dealt with basic research on the nature and constitution of the noncellulose wood components.

Last summer the Wood Chemistry Committee of TAPPI started to investigate if a fourth lignin symposium could be held in the summer of 1961. It then was recognized that the 18th International Congress of Pure and Applied Chemistry had scheduled a meeting in Montreal in August, 1961. The Wood Chemistry Division of this organization would have a three-day meeting there with basic research papers on the latest knowledge pertaining to the wood components. We decided it would be desirable to schedule a lignin symposium immediately subsequent to this Montreal meeting by selecting a theme which would *complement* rather than *duplicate* the preceding meeting. The subject area for this symposium was therefore designated to aspects of lignin knowledge relative to its utilization.

Many of us who are engaged in research in connection with lignin utilization have long felt the need for a forum where the practical aspects of lignin research and knowledge can be brought out for discussion and better understanding. By such planning, also with respect to time and geographical location, we hoped that many of those attending the Montreal meeting would find it possible to subsequently participate in the lignin symposium. This would provide the opportunity for basic research workers not only to teach and discuss newly acquired knowledge but also to learn about the many areas where further knowledge can contribute to progress in utilization. It is therefore gratifying that so many eminent lignin researchers are participating in this symposium. Indeed, several of those present have their names directly connected with specific lignin preparations which have advanced the understanding of the nature of lignin.

Some 100 years ago the acid and alkaline cooking processes were developed for liberating cellulose fibers from wood. In this way, 50% of man's most important and self-replenishing raw material has been converted into paper and other cellulose products of the highest significance for our civilization. It is an appalling fact that the other 50% of the tree is either burned up with inferior calorific efficiency or discharged into waterways with the hope that nature will dissipate its pollutional effect. There has not been a lack of research and technical effort to turn this waste of natural resources into economic importance, and yet deplorably few are the developments which have resulted in sound industrial production. Years ago a well known pulp and paper technologist made the cynical remark: "You can make about anything from lignin but money!" Sorry to say, this statement is largely true also today.

A few examples may touch on the nature of some of the problems. About 25 years ago the first plant production of vanillin was started up and is today—together with a few other plants—supplying the greatest part of the world's flavor market with lignin-vanillin. Basic research has informed us that lignin is made up of aromatic building stones, but aside from the low yield, expensive conversion to vanillin, no cleavage reactions known today for production of monomeric aromatic compounds from lignin are sufficiently attractive commercially to supply intermediates for large industries, such as plastics, synthetic fibers, detergents, etc. Lignosulfonates are gradually finding numerous industrial applications as we learn about their physical properties, how to modify them and where to apply them. But such developments are largely empirical and handicapped by our incomplete understanding of the lignin macromolecule and its functional groups. We must today admit that we do not even have satisfactory quantitative analytical methods for determination of lignin and lignosulfonates. The trend towards increasing use of deciduous trees in the pulping industries has increased the problems of lignin utilization. Practical behavior differences between softwood and hardwood lignins are largely unexplained by today's fundamental concepts of the structure of these two lignins.

The pulping processes for obtaining cellulose fibers were developed empirically, certainly without much benefit from knowledge of wood chemistry. But as such knowledge has developed, it has been injected into the pulping technology.

TAPPI was organized in 1915 as a technical society of experts dedicated to the application of sciences in pulp and papermaking, and to disseminate and discuss knowledge and technical progress in this industry. An early attitude of secrecy and closely guarding of individually gained technical advances has been gradually dispelled down through the years. This evolution, by the recognition that receiving also requires giving, has yet to be developed in the lignin utilization area. With this symposium, we think we are actually pioneering in this attitude, as applied to by-product utilization know-how. We met with the characteristic reticence when we solicited papers for this meeting. The program chairman for this symposium had a difficult assignment. Dr. W. M. Hearon has carried this out with enthusiasm, competence, and energy, and he deserves the appreciation from the participants. It was sad to learn late in June that he was striken ill. While he is now well on the way to recovery, he will not be able to attend this symposium which he has worked so hard to organize. We will all miss him, and

Lignin Symposium banquet head table—R. G. Macdonald, TAPPI; W. Jensen, Finnish Pulp & Paper Research Institute; H. F. Lewis, The Institute of Paper Chemistry; Holgar Erdtman, The Royal Institute of Technology—Sweden; J. R. Salvesen, Marathon Div., American Can Co.; L. G. Stockman, Central Laboratory, Swedish Cellulose Industry; E. G. Locke, U. S. Forest Products Laboratory; and H. O. Teeple, TAPPI

send him our sincere wishes for his recovery, and continued success in directing utilization through wood chemistry.

We now appeal to the participants to contribute in the exchange of knowledge during the session discussions and individual group gatherings. This will make for the success of the symposium, provide interest for a subsequent one—and make the task of organizing it easier!

Program

All of the technical sessions were very well attended and the subjects of the papers presented provoked substantial discussion from the audience.

A great highlight of the symposium was the banquet address by Dr. Holger Erdtman of the Swedish Royal Institute of Technology whose subject was "A Chemist Looks at the Forest."

Aided by his charming wife who manipulated two slide projectors, Dr. Erdtman captivated his audience for one and one-half hours with a most absorbing, provocative, and inspirational discussion of the things a chemist sees when he looks at a forest.

In addition to the excellent papers presented at the symposium, the titles, authors and abstracts of which are listed below, H. F. Lewis, The Institute of Paper Chemistry, and chairman of the TAPPI Development and Research Division conducted a panel discussion on the subject of "Where Do We Go From Here."

The four panelists, listed below, discussed the several subjects shown respectively.

G. H. Chidester, Forest Products Laboratory, Madison, Wis., "The Utilization of Lignin in Pulp."
R. E. Barnwell, Robeson Process Co., Erie, Pa., "The Future for Isolated Lignin as in Spent Liquors."
I. A. Pearl, The Institute of Paper Chemistry, Appleton, Wis., "The Future in Making Pure Chemicals from Lignin."
A. Bjorkman, Laboratoriet for Almen Teknisk Kemi, Danmarks Tekniske Hojskole, Copenhagen, Denmark, "Lignin as a Potential Raw Material for Destructive Distillation, etc. and its Future."

Abstracts

1. The Separation of the Components in Spent Sulfite Liquor," by W. Jensen, K. E. Fremer, and K. Forss, Finnish Pulp and Paper Research Institute, Helsinki-Helsingtors, Finland.

One of us (K. F.) has shown that the aromatic components of spent sulfite liquor can be separated from the nonsulfonated hemicellulose components by means of ion exclusion chromatography. The lignosulfonic acids separated as two partly overlapping peaks. Analyses showed that all the lignosulfonic acids in the liquor had identical properties, which in addition are typical for so-called α lignosulfonic acids. In addition sulfonic acids greatly resembling the lignosulfonic acids in composition as well as nonaromatic sulfonic acids were partially separated.

In view of the above results, it was of interest to attempt a more effective separation of the lignosulfonic acids and the other mentioned sulfonic acids. As gel filtration was expected to be a suitable method for this purpose, experiments were carried out using "Sephadex," a polymeric substance prepared in different porosites by cross-linking the polysaccharide dextran. As this substance contains no appreciable amounts of ionizing groups, a column containing "Sephadex" functions primarily as a molecular filter.

In experiments with columns of Sephadex it was found that the sulfonic acids of spent sulfite liquor could be more effectively separated from each other by gel filtration than by ion exclusion.

As Sephadex is unable to effect separations by ion exclusion it cannot separate strong and weak electrolytes as effectively as a cation exchange resin. As a consequence of this the monosaccharides in the spent sulfite liquor emerged from the Sephadex column as a sharp peak which was partly superimposed upon the last of the peaks containing the ultraviolet-absorbing components of the liquor.

2. "Determination of Reducing Bodies in Lignosulfonates. Comparison between Phenol and Swedish Methods," by J. Giguere, Lignosol Chemicals Limited, Quebec, Que.

A colorimetric method has been adapted to the quantitative determination of reducing bodies in lignosulfonates. These substances give an orange-yellow color when they are treated with phenol and concentrated sulfuric acid. The reaction is sensitive and the color stable. A comparative study was made between the phenol method and the Swedish method which is volumetric. A relation was found between the two methods and a factor was used as a correction for the color developed by lignin with sulfuric acid. According to a statistical analysis of the two methods, one is as good as the other when followed by a same operator, while the phenol method is more precise when used by many operators.

3. "Factors Affecting Evaluation of Dispersing Agents," by M. A. Baum, E. E. Harris, and R. F. Benning, Marathon Div. of American Can Co., Rothschild, Wis.

Session I—J. S. Barton, Crown Zellerbach Corp.; W. Jensen, Finnish Pulp & Paper Research Institute; J. Giguere, Lignosol Chemicals, Ltd.; M. A. Baum, Marathon Div., American Can Co.; and J. Benko, Sulfite Pulp Mfgrs. Research League

One of the major areas of utilization for lignosulfonates is as dispersing agents. Since lignosulfonates can vary widely in physical and chemical characteristics as well as in purity, and since such variations will profoundly affect the surface active properties, it is important that dispersing action is correctly evaluated with regard to the intended applications.

Dispersing agents are evaluated by measuring their effect on the rheological properties of the dispersed medium. Several factors must be considered when comparing different agents. Most important are: concentration of agent and of suspended particles, pH, time, temperature, and shearing stress that the system is subjected to, contamination of system, and particle size of suspended particles. Data are given to illustrate the effect and magnitude of these factors.

4. "The Effect of Molecular Size on Surface Active Properties of Lignosulfonates," by J. Benko, Sulfite Pulp Manufucturers' Research League, Appleton, Wis.

A new method for determination of relative molecular weight of soluble lignin compounds based on diffusion constants has been previously described. Employment of this method over a period of two years has made possible a more close evaluation of the effect which molecular size of lignosulfonates has upon certain of the surface active properties of the lignosulfonates. Data are presented correlating molecular size with dispersing effect upon kaolin clays. In general, the dispersion property increases with molecular size. Further studies in the area of adhesive and binding properties also show increasing value with increase in molecular size. Data are presented for binder properties when used with clay and sandy soils.

5. "New Utilization Prospects of Sulfite Liquor in the Federal Republic of Germany," by W. H. Gloz, Chemische Werke Zell-Wildshausen G.m.b.H., Division of Aschaffenburger Zellstoffweke AG Düsseldorf, Germany.

The West Germany wood pulp production is based almost entirely on the sulfite process which, besides wood pulp, produces some 600,000 to 700,000 tons spent cooking liquor solids. Only a fraction of the latter is presently utilized in other industries. Ever increasing pressure for reduction of stream pollution, together with economic requirements for more complete utilization of raw material, makes processing and marketing of the spent sulfite liquor solids important for the future of the German pulp industry. The following areas presently represent the largest and most promising new outlets to increase utilization: (1) coal briquetting and (2) agglomeration of ore and blast furnace dust.

Session II—A. S. Gregory, Weyerhaeuser Co.; W. H. Gloz, Chemische Werke Zell-Wildshausen, G.m.b.H.; I. A. Pearl, The Institute of Paper Chemistry; C. W. Dence and K. V. Sarkanen, State University College of Forestry, Syracuse University; and O. Ellefsen, Central Institute of Industrial Research—Norway

Session III—G. H. Tomlinson II, Howard Smith Paper Mills, Ltd.; J. W. T. Merewether, Ivanhoe, Victoria, Australia; P. R. Wiley and Joseph Marton, West Virginia Pulp and Paper Co.

Present status of these applications for spent sulfite liquor solids is discussed.

About one third of the spent sulfite liquor in Germany results from beechwood. Since beechwood spent liquor has different properties than that from softwoods, this complicates the problem, and a discussion of this and the present research efforts and aspects are presented.

6. "Studies on the Chemistry of Aspenwood. XIV: The Ether-Soluble Portion of Spent Sulfite Liquor from Authentic Quaking Aspenwood," by I. A. Pearl, D. L. Beyer, and O. Justman, The Institute of Paper Chemistry, Appleton, Wis.

Peeled authentic quaking aspen (*Populus tremuloides*) logs free of bark and cambium were pulped with an ammonia-base acid sulfite cooking liquor, and the resulting spent sulfite liquor was concentrated and extracted exhaustively with ether. The ether extract was fractionated by paper chromatographic, column chromatographic, and solvent distribution procedures, and the following compounds were isolated and identified: vanillin and syringaldehyde; benzoic, *p*-hydroxybenzoic, vanillic, and syringic acids, myristic, palmitic, stearic, oleic, linoleic, and linolenic acids; C_{25} and C_{26} saturated long chain aliphatic alcohols; several isomers of lirioresinol; three flavonoid coloring matters; and an organo-silicon compound. In addition, a number of other compounds were indicated, but not identified. The occurrence and significance of the identified materials are discussed.

7. "The Oxidation of Lignin Model Compounds and Wood Meal with Chlorine Dioxide and Sodium Chlorite," by C. W. Dence and K. V. Sarkanen, State University College of Forestry, Syracuse University, Syracuse, N. Y.

Various lignin model compounds, together with birch and sprucewood meal, were reacted with aqueous solutions of chlorine dioxide and sodium chlorite. The following reactions were characteristic of the model compounds:

1. Rupture of the aromatic nucleus to form derivatives of the muconic acid methyl esters. The structures of the latter were conclusively demonstrated by elemental analysis, conversion to known compounds and by infrared spectroscopy. The ester groups of these compounds were unusually sensitive to alkaline hydrolysis at room temperature.
2. The oxidation of the side chain resulting in the formation of methoxybenzoquinone and chloromethoxybenzoquinone.

The nonquantitative recovery of reaction products by ether extraction indicated that at least some of the reaction intermediates were susceptible to further oxidative and/or hydrolytic degradation.

Session IV—F. J. Ball, West Virginia Pulp & Paper Co.; J. Grierer, Swedish Forest Products Research Laboratory; T. Enkvist, University of Helsinki, Helsingfors; and D. W. Goheen, Crown Zellerbach Corp.

The rates of decomposition of vanillyl and veratryl alcohols in chlorine dioxide were followed determining the amount of methoxyl groups recoverable as methanol at various time intervals. The demethylation may be due to the direct action of chlorine dioxide and sodium chlorite on the model compounds or by hydrolytic action on the esters of the muconic acid derivatives. With both alcohols there was an initial rapid rate of methanol formation followed by a slower one. The final recovery of methoxyl groups as methanol was 85–90% of the theoretical value. A parallel situation was observed with the birch and spruce wood meal except that the final yields of methanol were 35–45%, respectively.

The relation of these findings to the commercial delignification and bleaching processes is discussed.

8. "IR-Investigations of Lignin—A Discussion of Some Recent Results," by S. Kolboe, The Norwegian Pulp and Paper Research Institute, Oslo, Norway and O. Ellefsen, Central Institute of Industrial Research, Oslo, Norway.

The IR-investigations to be dealt with in this paper were planned in order to get information about the possibilities that IR-studies may provide when applied in connection with structure problems in wood chemistry. The experiments have so far only been performed on samples of Norwegian Spruce.

Because of the fact that some authors have recently expressed the opinion that the lignin content in wood is much lower than hitherto believed, it was thought of interest to estimate this amount by independent methods. Such an independent estimation may be procured by means of IR-spectroscopy. The result obtained by this method was found to be in agreement with the ordinarily accepted value, viz. 28%.

The investigations have mainly been concentrated on Brauns lignin, Björkman lignin, and lignin in the original nontreated wood as it is revealed by IR-difference spectra. Judged from such a comparison it seems that only small structural changes are occurring in the lignin during the isolation carried out according to the methods of Brauns and Björkman.

From the details in the IR-spectra it has been tried to obtain information on the amount of carbonyl groups and phenyl coumaran groups in the lignin. The results obtained from this part of the investigation were: approx. 0.15 CO/-OCH_3 and less than 0.05 phenyl coumaran groups per one coniferyl alcohol group.

By deuteration it was finally found that the OH vibration frequency is mainly located in the region 1350 cm^{-1}.

9. "The Precipitation of Lignin from Eucalyptus Kraft Black Liquors," by J. W. T. Merewether, Ivanhoe, Victoria, Australia.

For satisfactory operation of a commercial lignin plant it is essential that the precipitated lignin be readily separable from the acidified black liquor. This can be achieved either by coagulation of the precipitate to a tar or by filtration. In this paper are described the results of a series of investigations of the conditions under which lignin salt is precipitated from eucalyptus kraft black liquor, using both carbon dioxide and sulfuric acid for acidification.

For separation by coagulation and using carbon dioxide for acidification, it was found that the pH of acidification is extremely important. Although the yield or precipitated lignin salt increases as the pH drops, stepwise carbonation and coagulation showed that only the lignin salt precipitated above pH 9 could be coagulated. Increasing the concentration of the black liquor led to an increase in the yield or coagulated lignin salt, while ageing or oxidation of the black liquor resulted in a decreased yield. A comparison of carbon dioxide and sulfuric acid as acidifying agents indicated no difference, each giving the same yield of apparently identical product.

For separation by filtration, and using sulfuric acid for acidification, it was found that a filtrable product results when black liquor is acidified at a temperature just below the coagulation temperature of the lignin salt. The filtration rate increases with temperature up to this point, after which it decreases. Increasing the concentration and decreasing the pH both gave pronounced increases in yield.

Conversion of lignin salt to lignin is also carried out most effectively with respect to filtration rate at a temperature of about 80°C. Filtration in this operation is also improved by increasing the concentration up to 20–25%, this giving both faster filtration and a drier filter cake.

10. "Development of Useful Products from Kraft Lignin," by P. R. Wiley, West Virginia Pulp and Paper Co., Charleston, S. C.

The 6,500,000 tons of kraft lignin estimated to be available from the kraft pulping industry per year constitutes the largest source of raw lignin available in this country. Accordingly, in considering commercial utilization of lignin, more emphasis on kraft lignin as opposed to other lignins is indicated. The structural features of kraft lignin which make it susceptible to sulfonation, esterification, etherification, salt formation, aldehyde, condensation, halogenation, and other reactions are well known. However, the relative quantities of active groups, as well as the average molecular weight of the lignin, varies depending on wood source and processing conditions.

By intensive work, however, standardized derivatives have been developed from raw kraft lignin starting material. The author's company has spent over $5,000,000 in a 20-year program in developing reproducible and useful products from kraft lignin as such and depend on consumers for application research, the company found it necessary to acquire personnel trained in the major application technologies and study the applications intensively. Though costly and time consuming, this program has led to current uses for lignin derived products of two main types. The first, involving the surface active properties of water soluble salts derived from lignin, includes emulsion stabilization, cement processing, ceramic processing, dye dispersion, insecticide dispersion, pigment dispersion, and others. The second type of use, in which the lignin derived product becomes integral with the end product in large percentage, includes the use of newly developed products in resins and in rubber. Although the above developments are far from consuming the total raw kraft lignin available, they demonstrate what can be done by the application of time and money to lignin as a raw material.

Session V—D. L. Brink, University of California; and M. Oshima and K. Kashima, The Noguchi Institute

11. "Lignin and Plastics," by K. Kratzl, University of Vienna and Austrian Wood Research Institute, Austria.

Lignin may be used as a raw material for plastics in two principally different ways: (1) degradation to low molecular compounds which, after separation, can be polymerized to plastics. (2) utilization of the high molecular lignin itself as a component in copolymerizations and cocondensations.

The first way is difficult and expensive but in well known reactions valuable products can be obtained as there are phenol plastics, epoxide resins and guacyl styrene polymers. The second way is simple and inexpensive but yields products of minor quality. According to biological reasons lignin shows a certain pattern of condensation which is altered during the technical process of separation, for example during pulping or hydrolysis.

This paper deals especially with the second way. The utilization of lignin in the phenol resins industry and its reactions with isocyanate are discussed. The author has developed a method to determine the degree of condensation which is present in the lignin after various pretreatments during technical processes. This method, a quantitative analysis of oxidation products, is discussed in detail. Thereby an obvious parallelism between degradability and utilization in the phenol plastics industry could be observed in case of hydrolysis lignins. Hydrolytical treatment of wood (water, diluted or concentrated acids which are used during technical processes) shows certain phenomena which could be interpreted as an acidolytical disproportionation. In acid media this reaction goes parallel with the real cocondensation with the plastics. The position of the condensed degradation products, e.g., parahydroxy- or orthohydroxy benzoic acids, if lignins are used which are labeled with radiocarbon at various positions of the side chain. The behavior of hardwood lignins during self condensation is investigated. Thereby an embarrassing parallelism between the degradability of syringe units at the one hand and of vanillin units at the other could be observed.

The reaction of lignin with isocyanates and di-isocyanates has been investigated by means of 20 various model compounds. The results of an addition polymerization of hydrolysis lignins with isocyanates is discussed briefly. No products of technical value could be obtained since the number of active positions is too low. Finally the properties of the active positions in lignin, which may undergo copolymerization, are discussed. Methods to increase the number of active positions are reviewed.

12. "The Degradation of Lignin by Alkali," by J. Gierer and I. Kunze, Swedish Forest Products Research Laboratory, Stockholm, Sweden.

As a part of our investigation on the structural changes of lignin during alkali and sulfate cooking, a study of the main degradation reaction (the alkaline hydrolysis of aryl alkyl ether bonds) was initiated.

The results hitherto obtained show that the cleavage of such bonds under conditions usually employed (2 hr. heating at 170°C. in 2*N* NaOH) requires the presence of a neighboring hydroxyl group in the propane side-chain. These findings are easily explained by the assumption that the splitting reaction proceeds via an epoxide intermediate. Experimental support for the validity of this theory is provided.

The elucidation of the main course of the alkaline cleaving reaction (and further experiments based on the above results) may contribute to a better understanding of the structural requirements of the alkaline scission and also to better knowledge of the chemical properties of lignins which have been subjected to a treatment with alkali.

13. "The Demethylation and Degradation of Lignin or Spent Liquors by Heating with Alkaline Reagents," by T. Enkvist, J. Turunen, and T. Ashorn, University of Helsinki-Helsingfors, Finland.

Continuous or batch process pressure heatings of kraft or sodium-base sulfite spent liquors with addition of restricted amounts of sodium sulfide and sodium hydroxide at 250–285°C. give high yields of ether soluble material. The other solubles are shown to consist of demethylated and reactive phenols such as pyrocatechol, protocatechualdehyde and acetopyrocatechol in addition to nonvolatile and volatile acids, methyl sulfide and methyl mercaptan (Th. Ashorn).

The yields and the composition of the reaction products are very dependent upon the reaction conditions. The temperature, heating time and water contents of the reaction mixture are of particular influence in this respect. Corresponding results are obtainable at ordinary pressure by the use of high alkali concentration. High yields of the ether soluble substances can also be obtained by the treatment of sulfate lignin with lime and super-heated steam (J. Turunen).

Methods for the chromatographic analysis of the obtained reaction products have been studied.

14. "Sulfur Chemicals from Lignin," by W. M. Hearon, W. S. MacGregor, and D. W. Goheen, Crown Zellerbach Corp., Camas, Wash.

The reaction of sulfidic sulfur with the methoxyl groups of kraft lignin produces methyl mercaptan (MM) which in turn can react with additional methoxyl groups to form dimethyl sulfide (DMS). A commercial plant to produce DMS and MM using this process has been operating since the first of the year at Crown Zellerbach's Bogalusa, La., mill. Varying the reaction conditions can produce different proportions of MM and DMS, the total yield however runs about 2.6% of the kraft black liquor solids.

This process applied to all kraft production in the United States could produce about 400,000 tons of sulfur chemicals per year. Present markets for DMS are limited mainly to gas odorants and for MM to the manufacture of methionine which equal only a small fraction of the quantities which could be made.

As a means of expanding markets for DMS and MM, a number of derivatives have been studied which could be readily prepared from the two basic sulfur chemicals.

Dimethyl sulfoxide (DMSO) can be made from DMS by oxidation and the probably most practical process uses oxygen and nitrogen dioxide as a catalyst, liquid phase. A plant at Bogalusa, adjacent to the DMS plant, has been operating on this process since Jan. 1, 1961. DMSO is a powerful solvent and as such is finding many applications.

Further oxidation of DMSO gives dimethyl sulfone ($DMSO_2$) which is an odorless solid. The best process seems to be via nitric acid with recovery and reuse of the nitrogen oxide gases. An alternative process proposed by M. W.

Lignin Symposium Panel—H. F. Lewis, The Institute of Paper Chemistry; G. H. Chidester, U. S. Forest Products Laboratory; A. Bjorkman, Tekniske Hojskole—Denmark; I. A. Pearl, The Institute of Paper Chemistry; and Robert Barnwell, Robeson Process Co.

Kellogg Co. uses osmium tetroxide as a catalyst to disproportionate DMSO and DMS and the sulfone. Possible applications for $DMSO_2$ include as a plasticizer and selective solvent for petroleum.

Chlorination of DMS produces a number of products depending on the degree of chlorination. Some of these derivatives may have application as agricultural chemicals or intermediates.

Ternarization of DMS produces products which may have application as antimycotics.

MM on mild oxidation is converted to dimethyl disulfide (DMDS). A simple process for this conversion employs air, vapor phase, over a copper catalyst supported on carbon. DMDS is an outstanding solvent for sulfur and as such has possible application for extraction of sulfur from ores or as a solvent to increase sulfur reactivity.

15. "Lignin Liquefaction by High Pressure Hydrogenation," by M. Oshima and K. Kashima, The Noguchi Institute, Tokyo, Japan.

The Noguchi Institute has succeeded in the liquefaction of lignin by high pressure hydrogenation with very high yield. Phenols are obtained as main components of the liquefied oil. On the basis of lignin 100 by weight, an example of material balance is as follows:

Light solvent fraction (below b. t. 100°C.)	5
Monophenol fraction (b. t. 190–230°C.)	46
Catechol fraction (b. t. 230–260°C.)	6
Heavy oil fraction (b. t. 260–320°C.)	18

Monophenol fraction consists of para-cresol, ortho-cresol, ethylphenol, propylphenol. Catechol fraction consists of pyrocatechol and its derivatives. In other words, almost all components of our liquefied oil are phenols without methoxyl radicals. We must assume the new types of the reaction mechanism of lignin decomposition.

Acid lignin from wood hydrolysis, lignin and lignin sulfonic acid from waste liquor of sulfite pulp process, thio-lignin from waste liquor of sulfite pulp process, thio-lignin from waste liquor of kraft process and soda pulp lignin can be used as raw material of our process.

The continuous pilot plant, of which the treating capacity is 30 kg. lignin per day, was constructed in our institute in 1956 and very good results have been achieved. The larger pilot plant of 1000 kg. lignin per day is now under preparation, being subsidized by Japanese Government and the associate companies of our institute.

Our result implies that the possibility to industrialize lignin liquefaction by high pressure hydrogenation has remarkably increased.

Seventh Annual Pulp and Paper Industry Electrical Engineering Conference

The Seventh Annual Conference on Electrical Engineering as applied to the Pulp and Paper Industry, was held during June 21–23, 1961, in the Auditorium of the McCracken Hall on the extensive campus of Western Michigan University, located in Kalamazoo, Mich.

This conference was attended by some 150 engineers from the mills and allied industries. Attendance was from wide spread areas and included mill electrical engineers from as far north as Canada and as far west as Washington, as well as the southern and eastern areas represented by Florida and Massachusetts.

An interesting program of technical papers and timely addresses, directed at the improvement of technical operations in the pulp and paper mills, highlighted the 3-day conference. Inspection trips to the nearby paper mills and other nationally known firms, producing a variety of products such as pharmaceutical supplies, musical and fishing equipment.

The conference opened at noon, Wednesday, June 21, with a joint committee luncheon in the University President's dining room. This was followed by separate committee meetings of our AIEE Pulp and Paper Subcommittee and our good neighbors from the north, The Electrical Engineering Committee of Canadian Pulp and Paper Association.

During the AIEE Subcommittee meeting Wednesday, Atlanta, Ga., was selected as the location for the Eighth Annual Meeting, tentatively arranged for the third week in May, 1962.

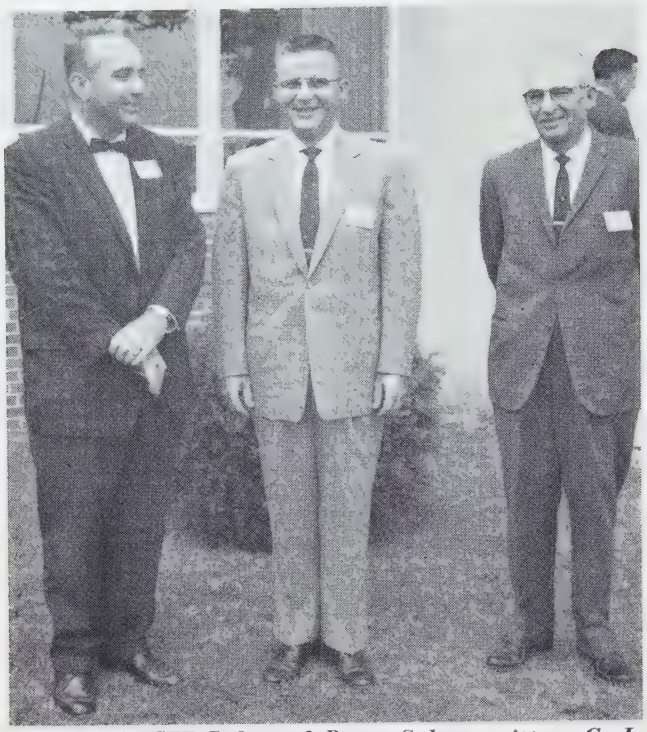
R. P. Derrick of Westinghouse Electric, Pittsburgh, Pa., was appointed program review chairman, along with W. S. Hines of Westinghouse, Atlanta, who will serve as local arrangement chairman. C. B. White of the Allis-Chalmers Mfg. Co., Norwood, Ohio, was appointed publicity committee chairman, and A. C. Cook, General Electric Co., Cincinnati, was appointed to serve with the Publicity Committee.

The technical aspect of the conference was introduced at 9:30 a.m., June 22 by D. B. Gearhart, General Electric Co., Kalamazoo, who was the local conference chairman for the subcommittee meeting. Mr. Gearhart introduced Dr. George E. Kohrman, dean of Applied Arts and Sciences, and Dr. John Fanselow, head of the Department of Pulp and Paper Technology, Western Michigan University, who welcomed those attending the conference on the University campus.

After a few opening remarks by Jesse M. Yarbrough, chairman of the subcommittee, Dr. Robert Diehm of Western Michigan University presented a very enlightening talk on "Engineering Developments In Papermaking." Dr. Diehm pointed out that lots of timber is going to waste faster than it is being used. He also pointed out that the helicopter could be used economically in logging many areas inaccessable for the conventional means of removal to the mill.

Papers delivered during the morning session were; "Techniques In Data Logging," by Robert P. Derrick, Westinghouse Electric, Pittsburgh, "System Components and Assembly," by W. Mikelson, General Electric Co., Erie, Pa., and "Applications" (data logging) by H. S. Ogelsby, St. Regis, Jacksonville, Fla.

During the Data Processing Seminar, Mr. Derrick discussed the types and methods of data logging along with the many instruments now available or under design, which may be utilized in measuring paper machine variables. Mr. Derrick stated these variables may be "read out" periodically in a data accumulating process. Mr. Derrick also discussed, "process computer control" systems involving calculated results from the measured variables. "These systems," Mr. Derrick stated, "are not prohibitively expensive and computers in general, should add eight and a half million people to the working force."



Officers of AIEE Pulp and Paper Subcommittee—C. L. Eletson, Hopper Paper Co., Program Review Committee; J. M. Yarbrough, The Mead Corp., Chairman, Pulp and Paper Subcommittee; J. S. Kirkland, Rome Kraft Co., Secretary, Pulp and Paper Subcommittee

Mr. Mikelson pointed out that the use of data logging and computer equipment is in an early stage of development and that the terminology used is not yet standardized. He stated also that "the computer is really simple, and when compared with human ability, has the capability of a fourth grader." Mr. Mikelson pointed out that the large scale memory or storage capacity of the computer is used to great advantage, due to the speed at which it may be utilized in scanning its memory. Mr. Mikelson also pointed to the many new techniques utilized in computer systems.

Mr. Ogelsby, in his discussion of "Applications," pointed out that rising costs in the paper industry are being compared to selling prices, but not at the same rate. He pointed out also that a cautious approach should perhaps be made in the automation of the industry.

The Data Processing Seminar was extended into the afternoon session which followed an enlightening luncheon speech by Dr. John Cruze, director of Summer School at WMU.

Dwain Bates of Potlatch Forest presented a résumé of the study of an IBM computer application of their Lewiston operation. In his résumé, Mr. Bates pointed to the fact that the purpose of this study is to predict quality during operation and to minimize off grade material. Mr. Bates stated that he does not believe data processing should be initiated "without specific purpose and an organized set of data."

In a question and answer session immediately following the seminar, dollar figures of $50,000 up for data logging and $100,000 up for computer analysis were mentioned.

The afternoon session with Charles Eletson, of Hopper Paper Co., Alton, Ill., presiding, was ended after the presentation of a paper on "Boiler Flame Out Control" given by P. K. Ryder of The Electronics Corp. of America.

The evening session followed a social hour, highlighting a humorous account of "Bends and Breaks," by Rice Kello of Investers Division Services, Evansville, Ind.

The third day of the conference began with the presentation of a paper on "The Application of Instantaneous Trip Circuit Breakers in Motor Control Centers," by C. Rice of Allen Bradley Co., Milwaukee, Wis. Mr. Rice discussed the magnetic trip circuit breaker as it is used in "combination" type starters when coordinated with the magnetic starter to provide short circuit and over current protection for electric motors.

A panel discussion on "D. C. Motor and Generator Insulation" prompted a very interesting question and answer discussion between a panel of experts, representing paper mills and electrical manufacturers, and the audience. The moderator for this panel was J. S. Kirkland, Rome Kraft Co., who is secretary of the subcommittee and representatives on the panel were:

D. E. Bivens, Jr., Olin Mathieson Chemical Co., West Monroe, La.
H. Murdock, Allis-Chalmers Mfg. Co., Norwood, Ohio.
P. T. Schuermann, General Electric Co., Schenectedy, N. Y.
A. G. Posluszny, Westinghouse Electric, Buffalo, N. Y.
W. Stiffler, Reliance Electric and Engr. Co., Cleveland, Ohio.

This panel discussion resulted in agreement that the d.c. motor and generator is being steadily improved as a result of research and new development in the area of improved organic varnishes and protective coatings employed polyester and epoxy.

The three day meeting was terminated with conducted mill tours during the afternoon of Friday, June 23rd.

This Seventh Annual Conference was jointly sponsored by the Pulp and Paper Subcommittee, The Western Michigan Section of AIEE, and the Department of Paper Technology, Western Michigan University. A very successful conference was held on this Western Michigan University campus, as a result of the contribution by members of the general Conference Committee, the splendid cooperation of the University and individuals at the University, as well as a number of informative and interesting papers presented under the direction and guidance of many leading representatives of paper mills and electrical manufacturers.

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TAPPI NOTES

THREE MONTHS ago these pages of "TAPPI Notes" were devoted to a review of the Association's publication policies, particularly those involving the magazine. Brief reference was made then to the units of measurement used in reporting data. The correct use of these units is of such vital importance that the subject deserves fuller treatment at this time.

No one questions the necessity of publishing in *Tappi* data in units which are readily understood by our readers. Such selection is not always easy, however, since the units employed frequently vary not only among countries but also among segments of our own industry. Furthermore, such usage is never completely static. The gradual replacement of trade units, peculiar to certain grades of paper, by English or metric units suitable for the entire industry is evidence of this changing pattern.

In its Standard Testing Procedures, TAPPI has endeavored to prescribe units of measurement which recognize both the nomenclature of our advancing technology and the trade customs of our industry. It is hoped that units thus selected will merit world-wide acceptance, so that technical men everywhere can read and write a common language.

In a technical society with world-wide membership it is hardly surprising that there are some members who strongly advocate the adoption of the metric system of units throughout TAPPI Standard Testing Procedures. Although recognizing the long range advantages of this system, the Association believes that any abrupt and arbitrary decision requiring the use of metric units would be neither possible nor desirable.

The Association has stipulated instead that all units of measure specified in Standard Testing Procedures shall be in the metric system, wherever practical. Where not practical other units may be employed, but whenever possible the equivalent metric units shall be included in the reported results.

In all TAPPI publications the authors are required to use wherever possible, those units of measurement prescribed in the appropriate Standard Testing Procedures. In many instances these procedures permit units in both the English and metric systems.

Listed below are the preferred units of measurement to be used in all TAPPI publications. This list will help authors by indicating which unit is preferred in those cases where more than one is permitted in the corresponding Standard Testing Procedure.

P. E. NETHERCUT
Executive Secretary

Preferred Units of Measurement for Use in TAPPI Publications

FOR WOOD

Density (green and moisture-free)	grams per cubic centimeter (g. per cc.)
Dirt in chips	dirt index
Moisture	per cent of original (moist) weight
Other components of wood	per cent of moisture-free weight
Density (green and moisture-free)	pounds per cubic foot (lb. per cu. ft.)
Volume of cord-wood	128-cu. ft. cords, bark on or off
Weight of unit volume of chips	pounds per cubic foot under 225 lb. per sq. ft. (or other stated pressure

FOR COOKING LIQUORS

Sodium salts	grams per liter (g.p.l.) or the equivalent calculated as g. Na_2O per l., for mill reports also as lb. Na_2O per cu. ft.
Solids content of liquors	grams per liter (g.p.l.), for mill reports also as pounds per cubic foot.
Density of liquors	grams per milliliter (g. per ml.), for mill reports also as degrees Baumé (Bé)
SO_2 and sulfites in liquids	grams per 100 ml. or grams per liter
Sulfur burner gas (SO_2 and SO_3)	milliliters per 100 ml. of dry gas (% by volume) or grams per liter.

FOR PULP

Airdry pulp	90 parts moisture-free pulp plus 10 parts water
Brightness	per cent based on MgO = 100
Classification	per cent by weight; specify instrument, screens, charge, running time and rate of flowthrough of water
Coarseness	decigrex, dg.—(mg. per 100 m.)
Viscosity	centipoises of solution having a specified concentration
Concentration (or consistency) of dilute suspension	grams per liter of suspension
Dirt	parts of equivalent black area of dirt per million visible on the outer surfaces of sheets of pulp.
Drainage time (board stock)	seconds for 10.6 g. stock at 80°F. to drain in the TAPPI SFMC drainage tester

Table I. Multiplying Factors to Convert Pulp Test Data to and from TAPPI Standard T 220 m

Metric units (TAPPI Standard)		*English units* (24 × 36—480)	(24 × 36—500)	(25 × 38—500)	(25 × 40—5)
Basis weight (g.s.m.)	to Std.	1.695	1.627	1.480	1.406
	from Std.	0.590	0.614	0.676	0.711
Density, g./cc. (water = 1)	to Std.	0.0667	0.0640	0.0583	0.0553
	from Std.	15.0	15.62	17.16	18.07
Bulk, cc./g. (water = 1)	to Std.	15.00	15.62	17.16	18.07
	from Std.	0.0667	0.0640	0.0583	0.0553
Burst factor (g. cm.$^{-2}$/ g.s.m.)	to Std.	0.415	0.432	0.475	0.500
	from Std.	2.41	2.31	2.10	2.00
Tear factor (100 × g. tear one sheet per g.s.m.)	to Std.	59.0	61.4	67.5	71.1
	from Std.	0.01605	0.0163	0.0148	0.0145
Breaking length (m.) Normally calculated to meters on any basis.					

Examples: (1) 60 g.s.m. × 0.711 = 42.7 lb. (25 × 40 − 500). (2) 12.0 "density" (lb. 25 × 40 − 500–thickness in mils) × 0.0553 = 0.644 g per cc. (3) 1 "point per lb." (24 × 36—480, or "100%") = 100 × 0.415 metric units. (4) 2.00 "tear ratio" (24 × 36—500) = 2 × 61.4 = 128 metric units.

Occasionally it is not possible to convert experimental data to units given above. If so, *all* the conditions for the text, i.e., type of sheet machine, basis weight, pressing conditions, etc., must be given so that the values reported are unambiguous and understandable.

Drainage time.....seconds for 1.2 g. moisture-free (pulp) pulp forming a 60 g.s.m. sheet to drain at 20°C. in the TAPPI Standard sheet machine

Fiber length.....millimeter—weighted average length by weight (if for whole tracheids only, the numerical average length in millimeter is acceptable provided that the lower length limit of fibers included in the count is stated.)

Freeness, C. S.....milliliters with 3-g. moisture-free specimen

Moisture.....per cent of original (moist) weight

Other components.....per cent of moisture-free weight of pulp

Specific surface.....square centimeter (or sq. m.) per gram of moisture-free pulp

Viscosity.....centipoises of solution having a specified concentration

For Pulp Test Sheets:

Unless otherwise stated in unavoidable cases only, it will be assumed that the sheets were made, conditioned and tested in accordance with the appropriate TAPPI Standards; also that the derived results are given as ratios of the *moisture-free* basis weight.

Note: In the case of paper (qv.) the ratios are calculated on the *conditioned* basis weight.

Basis weight (r).....grams per square meter (g.s.m.) moisture-free

Thickness (T or t).....0.001 in. = T (or microns = t); omit the Thickness and give the Bulk only.

Bulk (or specific volume).....cubic centimeters per grams (cc. per g.) (determined from the thickness of five sheets measured together under 7.5 p.s.i.)

Burst factor.....(grams per square centimeter) per (grams per square meter) g.cm.$^{-2}$/ g.s.m.

Tensile breaking load (p).....kilograms per 15-mm. strip width —preferably omit and give the Breaking length only.

Breaking length.....metric = 200,000 $p/3r$ (meters)

Density.....grams per cubic centimeters (g. per cc.)

Fold.....number of double folds on MIT or Schopper type instrument—specify which

Stretch.....per cent of original length

Tear (e).....grams force to tear single sheet—preferably omit and give Tear factor only.

Note: e = 16/5 of reading on the usual instrument when 5 sheets are torn together through 43 mm.

Tear factor.....metric = 100 e/r

FOR PAPER

Unless otherwise stated, it will be assumed that the paper was tested at 50% R.H. and 73°F. and in accordance with the appropriate TAPPI Standard, and that any derived units are given as a ratio of the *conditioned* basis weight.

Note: In the case of pulp test sheets (qv.), the strength ratios are calculated on the *moisture-free* basis weight.

Acid-soluble iron.....parts per million (p.p.m.) of moisture free weight

Air permeability of paper.....seconds per 100 ml. air per square inch under pressure of 4.89 in. (12.2 cm.) water

Alpha-, beta-, or gamma-cellulose in paper.....per cent moisture-free weight

Amount of mineral coating.....per cent decoated paper and as grams per square meter (g.s.m.) of conditioned weight

Arsenic in paper.....parts per million (p.p.m.) of moisture-free weight grains per square yard

Ash.....per cent moisture-free weight

Basis weight.....grams per square meter (g.s.m.) *conditioned* and if desired, followed by the trade size and weight in pounds in parentheses.

Note: Lb. (25 × 40—500) is not a trade size and no longer is the TAPPI Standard size.

Bulking thickness.....decimals of inch

Bursting strength.....pounds per square inch (p.s.i.)

Copper number.....on 100 g. moisture-free cellulose

Density.....grams per cubic centimeter (g. per cc.)

Dirt in paper.....parts per million (p.p.m.) of equivalent black area on surfaces

Dry and wet tensile.....kilograms per 15 mm. width

Folding endurance.....number of double folds on MIT or Schopper type instrument—specify which

Moisture.....per cent original weight of specimen

Table II. Multiplying Factors to Convert Ream Basis Weights to and from Grams per Square Meter (TAPPI Standard)

	Trade size (500 sheets)	*Trade to metric*	*Metric to trade*
Blotting	19 × 24	3.083	0.324
Book	25 × 38	1.480	0.675
Bristol and Tag	22½ × 28½	2.195	0.456
Cardboard	22 × 28	2.282	0.438
Cover	20 × 26	2.704	0.370
Demy size	17½ × 22½	3.571	0.280
Former TAPPI Standard	25 × 40	1.406	0.711
News and wrapping	24 × 36	1.627	0.614
Paperboard	1000 sq. ft.	4.881	0.205
Tissue (Double Crown)	20 × 30	2.343	0.427
Writing and printing	17 × 22	3.760	0.266

Organic nitrogen	per cent moisture-free weight
Other components of paper	if by chemical analysis, percentage of the moisture-free weight; if by physical analysis, percentage of the conditioned weight.
Paraffin in paper	per cent conditioned weight
Paraffin wax absorptiveness	per cent of conditioned paper
Pentosans in paper	per cent of original moisture-free cellulose
Reducible sulfur	per cent moisture-free
Rosin in paper or paperboard	per cent moisture-free weight per cent conditioned weight
Specific volume	the ratio: cubic centimeter per gram-to-
Starch	per cent moisture-free weight
Tear	grams force to tear single sheet (the most common instrument is calibrated in grams to tear single sheet when 16 sheets are torn together)
Thickness	decimals of inch, under pressure of 7 to 8 p.s.i.
Titanium pigments in paper	per cent of moisture-free weight
Water absorption of bibulous paper	seconds to absorb 0.1 ml. water
Water absorption of paperboard	seconds to absorb 0.05 ml. water
Water absorptiveness of nonbibulous paper	grams per square meter (g.s.m.) conditioned weight
Water immersion of paperboard	increase in grams of a 6-in. (15.25 cm.) square specimen after 10 min. immersion (or other specified time)
Water-soluble acidity or alkalinity	per cent as SO_3 or Na_2O of moisture-free weight
Water-soluble sulfates or chlorides	per cent of moisture-free paper
Water vapor permeability	grams per square meter (g.s.m.) per 24 hr.
Zinc pigments in paper	per cent moisture-free weight

GENERAL

Analysis of water	parts per million (p.p.m.)
Blocking range of wax	degrees Fahrenheit, °F.
Moisture in materials	per cent on an "as received" basis
Other components of materials	per cent; grams per liter (g.p.l.) on an "as received" basis (unless specified as on a moisture-free basis)
Tensile strength of wax	pounds per square inch (p.s.i.)
Thermal conductivity	B.t.u./(sq. ft.) (hr.) (°F.) (in.)
Viscosity	centipoises at specified concentration and temperature (or g.-cm. of torque with a specified viscometer)
Wax melting points	degrees Centigrade °C.

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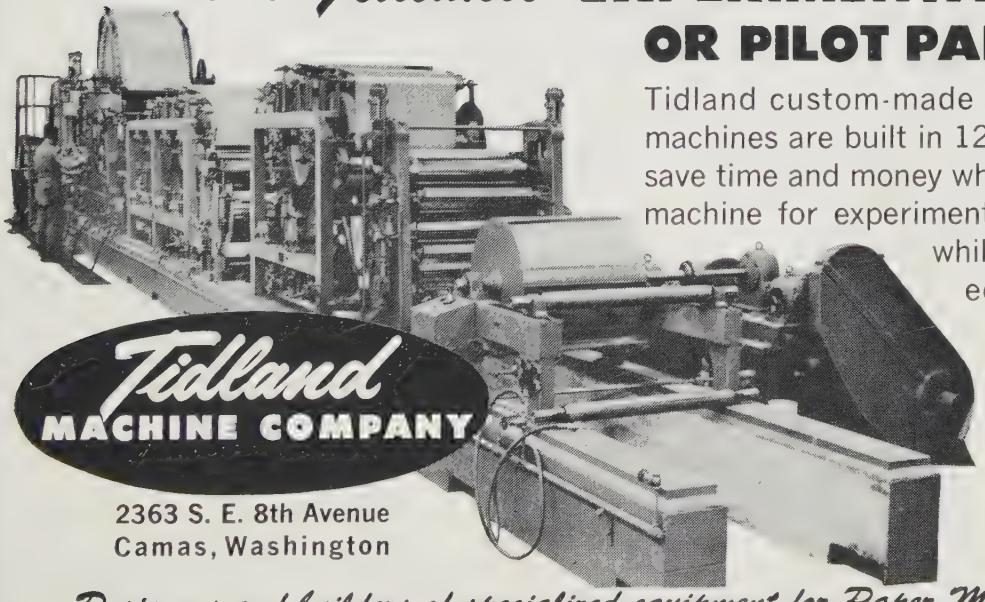

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TAPPI PROFILE

Lucien G. Rolland

Lucien G. Rolland, a member of the Executive Committee of the Association, is president and general manager of the Rolland Paper Company Limited, Montreal, Que.

Born in St. Jerome, Que., in 1916, he was educated at the College Jean-de-Brebeuf and Loyola College, Montreal, where he obtained a Bachelor of Arts degree. He then went to the Ecole Polytechnique of the University of Montreal and obtained a B.A. Sc., and C.E.

Since 1942, he has been with the Rolland Paper Company Limited, established in 1882 by J. B. Rolland, his great grandfather.

In that year he began his career in the Paper Industry as an engineer at Mont Rolland, Que., and in 1947 went to St. Jerome as Mill Manager. Shortly after, in 1949, he became Assistant General Manager for the company, and in 1952 Vice-President and General Manager.

He is now President and General Manager of the Rolland Paper Company Limited, with head office at 1645 Sherbrooke Street West, Montreal.

He has been a member of the Technical Association of the Pulp and Paper Industry since 1942 and was elected a member of its Executive Committee in 1958. Earlier this year, he was elected Chairman of the Executive Board of the Canadian Pulp and Paper Association. He is a director of the Bank of Montreal, the Royal Trust Company, Gatineau Power Company, General Security Insurance Co. of Canada, United Auto Parts Limited, Standard Life Assurance Company and Governor of the University of Montreal.

In addition, he is a member of the following associations: Corporation of Professional Engineers (P.Q.), Montreal Board of Trade, Canadian Red Cross Society (past president Quebec Division), Engineering Institute of Canada, and member of Council National Industrial Conference Board. He is a past chairman of the Quebec Division of the Canadian Manufacturers' Association.

In 1958 he was created Knight Commander of the Order of St. Gregory, and in 1960 he was awarded the degree of Doctor Commercial Science (Honoris Causa) by the University of Montreal.

Mr. Rolland, whose home address is St. Jerome Que., is married to the former Marie de Lorimier of Montreal. They have eight children, four sons and four daughters. His hobbies are hunting and fishing.

PERSONAL MENTION

New TAPPI Members

Acosta, Henry G., Methods Engineer, The Northwest Paper Co., Brainerd, Minn., a 1957 graduate of the University of North Dakota.

Agarwala, Prem M., Haindlische G. Papierfabriken, Augsburg, W. Germany, a 1956 graduate of Meerut College, Meerut.

Barney, Neal D., Paper Mill Engineer, Potlatch Forests, Inc., Lewiston, Idaho, a 1948 graduate of Purdue University.

Beecroft, George F., Joint Managing Director, Grove Mill Paper Co. Ltd., New Mills nr. Stockport, Cheshire, England, a 1939 graduate of Liverpool Technical College.

Blackburn, Robert H., Chief Librarian, University of Toronto, Toronto, Ont., Canada.

Blanpied, Robert H., Technical Sales-Service Representative, Hercules Powder Co., Holyoke, Mass., a 1957 graduate of Colorado College.

Boyce, William B., Research Engineer, Beloit Iron Works, Beloit, Wis., a 1959 graduate of the University of Wisconsin.

Brennan, Edmond P., Chief Chemist, National Board & Paper Mills Ltd., Granagh, Waterford, Ireland, a 1957 graduate of University College, Dublin.

Caceres-Rojas, Hernan, Chemist, Instituto Investigaciones Technologicas, Bogota, D.E., Colombia, a 1959 graduate of the National University.

Cantieri, William F., Vice-President I/c Engineering, Diamond Power Specialty Corp., Lancaster, Ohio, a 1935 graduate of Virginia Polytechnic Institute.

Cantini, Maurizio, Chief Production Engineer, Sterzi Paper Mill, Varese, Italy, a 1953 graduate of Politecnico di Milano.

Chandra, Partap, Process Engineer, Alliance Paper Mills Ltd., Merritton, Ont., Canada, a 1954 graduate of O-V-Miller Polytechnic Institute, Munich, Germany.

Charles, George E., Papermaking Research, Anglo Paper Products Ltd., Quebec City, Canada, a 1959 graduate of McGill University with a Ph.D. degree.

Clark, Robert E., Manager, Latex Development, Spencer Chemical Co., Merriam, Kansas, a 1942 graduate of Washington & Jefferson College.

Cowgill, Eric, Chemist, Technical Service Development, Brown & Polson, Ltd., Trafford Park, Manchester, Lancs., England, a graduate of Burnley Technical College.

Creighton, Richard L., Product Engineer, Union Carbide Corp., Silicones Div., Tonawanda, N. Y., a 1944 graduate of the University of Michigan.

Dadswell, Herbert E., Chief, Forest Products Div., Commonwealth Scientific & Industrial Organization, South Melbourne, Vic., Australia, a 1924 graduate of the University of Sydney, with a D. Sc. degree in 1941.

Do, Vy, Trainee, Otis Mill, International Paper Co., Chisholm, Me., a 1956 graduate of the University of Saigon, and a 1960 graduate of the University of Montreal.

Duckworth, Brian, Technical Sales Representative, Scapa Dryers, Ltd., Blackburn, Lancs., England, a 1954 graduate of Leeds University.

Duffy, James P., Manager, Technical Service Dept., HumKo Products, Chemical Div., Memphis, Tenn., a 1947 graduate of The Polytechnic Institute, Brooklyn.

Ebeling, Carlos H., Chief Engineer, Cia. Federal de Fundicao, Rio de Janeiro, Est. Guanabara, Brazil, a 1944 graduate of Colegio Novo Ateneu.

Ellis Anwyl, G., Technical Director, Walmsleys (Wigan), Ltd., Pagefield Ironworks, Wigan, England, a graduate of the Royal Technical College, Glasgow.

Epstein, Harry T., Treasurer and Chairman of the Board, Proven Paper Mfg. Co., Inc., Norwood, Mass.

Erickson, William H., Project Leader, Technical Dept., Waldorf Paper Products Co., St. Paul, Minn., a 1958 graduate of the University of Minnesota.

Farell C., Guillermo, Chemical Engineer, Papelera Iruffa Arcelus Hnos S.A., Mexico, D.F., a 1958 graduate of the University of Mexico.

Georgii, Ernst U., Paper Technologist, Sto-Chem., Ltd., Bromsgrove, Worcestershire, England, a graduate of Julius Maximillian University, Wurzburg, Germany.

Gordon, George, Project Engineer, Diamond National Corp., Stamford, Conn., a 1948 graduate of the University of New Hampshire.

Gusmer, John H., General Manager, Filter Materials, Inc., Waupaca, Wis., a 1954 graduate of Massachusetts Institute of Technology.

Guttman, Hans, Manager, Montana Grafica C. A., Caracas, Venezuela.

Herborg, Ina, Technical Librarian, Miehle-Goss-Dexter, Inc., Chicago, Ill., a 1935 graduate of the University of Wisconsin.

Hines, Wm. Stewart, Pulp and Paper Engineer, Southeastern Region, Westinghouse Electric Corp., Atlanta Ga., a 1946 graduate of the University of Michigan.

Humphrey, John B., Chemist, G. S. Ziegler & Co., New Market, N. J. Attended the College of the City of New York and Stevens Institute.

Johnson, Kenneth O., Research Chemist, Minnesota & Ontario Paper Co., International Falls, Minn., a 1958 graduate of Iowa State University.

Johnson, Ralph A., Jr., Assistant Chief Engineer, Oxford Paper Co., Rumford, Me., a 1943 graduate of the University of Maine.

Kamel, Hussein M., Managing Director, Al Nasr Company for Bagasse Pulp Industry, S.A.A., Cairo, Egypt, U.A.R., a graduate of the American University, Cairo, with a Ph.D. degree from Strasbourg University.

Kemper, Clarence A., President, Joseph Kaye & Co., Inc., Cambridge, Mass., a 1956 graduate of Massachusetts Institute of Technology.

Kochendorfer, Irwin, General Manager, Hermann Finckh G.m.b.H., Reutlingen/Wurtt., Germany, a 1931 graduate of Wurttemberg University.

Kresse, Herman J., Jr., Wax Technologist, Continental Oil Co., Houston, Tex., a 1951 graduate of Arkansas State Teachers College.

Leake, Preston H., Manager, Chemical Research, Albemarle Paper Mfg. Co., Richmond, Va., a 1954 graduate of Duke University with a Ph.D. degree.

Lee, Sangjai, Chemical Engineer, Korean Government Printing Agency Tae-Jon Paper Mill, Seoul, Korea, a 1946 graduate of The Seoul National University.

Lamarchand, Jean, Mill Manager, Societe Anonyme Papeteries Darblay, Grand Couronne, (Seine-Maritime) France, a 1935 graduate of Ecole Centrale Arts and Manufactures.

Manson, Dan W., Group Leader, Paper Making Research

Group, Oxford Paper Co., Rumford, Me., a 1934 graduate of the University of Vermont, with a Ph.D. from The Institute of Paper Chemistry in 1959.

Marzolo, Paolo, Technical Service Engineer, Societa Iniziative Cartarie-SICAR-S.p.A., Milano, Italy, a 1956 graduate of the University of Padova.

Meaker, Douglas W., Resin Group Leader, Riegel Paper Corp., Milford, N. J., a 1957 graduate of Haverford College.

Miller, Robert S., Librarian, J. J. Berliner & Staff, New York, N. Y., a 1959 graduate of the College of the City of New York.

Mitchell, Dewitt G., Sales Manager, Simonds Worden White Co., Dayton, Ohio, a 1948 graduate of Sinclair College.

Mona, George E., Technical Director, Special Fabrics Div., Sayles Finishing Plants, Saylesville, R. I. Attended Brown University.

Morton, John F., Chemist, Congoleum-Nairn, Inc., Cedarhurst, Md., a 1957 graduate of Lehigh University.

Nagaoka, Mikio, Chief, Designing Dept., The Kure Shipbuilding & Engineering Co., Ltd., Kure City, Japan, a 1941 graduate of Tokyo University.

Nelson, John S., Salesman, American Marietta Co., Seattle, Wash., a graduate of the University of Washington.

Orcutt, William P., Technical Superintendent, Consolidated Water Power & Paper Co., Wisconsin Rapids, Wis., a 1956 graduate of the New York State College of Forestry.

Padova, Giuliano, Vice Director de Fabrica, Papelera Rio Parana, Buenos Aires, Argentina.

Partch, John G., Director, Chemical Development, The North American Coal Corp., Cleveland, Ohio, a 1942 graduate of Miami University.

Phillips, Joshua, Commercial Development, Nalco Chemical Co., Chicago, Ill., a 1957 graduate of Williams College.

Pierce, John F., Jr., Graduate Student, School of Industrial Management, Massachusetts Institute of Technology, Cambridge, Mass., a 1961 graduate of Harvard University.

Pool, Michael C., Plant Manager, Wood Pulp Bleach Plant, Courtaulds, Ltd., Flint, Wales, U.K., a 1946 graduate of Imperial College.

Ptak, Louis R., Laboratory Director, Continental Can Co., Inc., Chicago, Ill., a 1938 graduate of the University of Iowa.

Rabbi, Renzo, Chemist, Sterzi Paper Mill, Varese, Italy, a 1939 graduate of the University of Modena.

Rooney, Joseph E., Chemist, Dierks Paper Co., Pine Bluff, Ark., a 1958 graduate of the University of Houston.

Rusten, Dag, Assistant Mill Superintendent and Head of Laboratory, Mjondalen Cellulose A/S, Mjondalen, Norway, a 1961 graduate of The Technical University of Norway with a Ph.D. degree.

Salzberg, Harold K., Section Head, Casein Research and Development, The Borden Chemical Co., Bainbridge, N. Y., a 1927 graduate of the University of Wisconsin with a Ph.D. degree.

Sellers, Jeff C., Jr., Section Engineer, Gulf States Paper Corp., Demopolis, Ala., a 1952 graduate of Auburn University.

Serrano, E. H., Head Librarian, Scientific and Technical Documentation Center of Mexico, Mexico, D.F.

Slavin, William, Associate Partner and Chief Engineer, Fellheimer & Wagner, New York, N. Y., a 1949 graduate of the Polytechnic Institute of Brooklyn.

Stone, James N., Chemical Engineer, Product Development, Organic Chemicals Dept., The Glidden Co., Jacksonville, Fla., a 1947 graduate of Vanderbilt University.

Strauss, Roger W., Manager, Research and Development, Nekoosa-Edwards Paper Co., Port Edwards, Wis., a 1949 graduate of the New York State College of Forestry with a Ph.D. degree in 1961.

Takashima, Naoichi, Manager, Research and Development, Mitsubishi Petrochemical Co., Ltd., Yokkaichi, Mie-Ken, Japan, a 1939 graduate of Tokyo Technical Institute.

Ulrich, Mrs. Olga, Chief, Research Dept., Zagrebacka Tvornica Papira, Zagreb, Yugoslavia.

Votava, Robert J., Research Engineer, Nekoosa Edwards Paper Co., Port Edwards, Wis., a 1958 graduate of the University of Wisconsin.

Walter, Heinz, Editor, Chemisches Zentralblatt, Berlin N 4, Germany, a graduate of Berlin University with a Pharm.D. degree.

Warner, Frederic L., Director of Sales, Paper and Foil Coatings Dept., Interchemical Corp., Newark, N. J., a 1936 graduate of Denison University.

Watson, William E., Technical Director, New Haven Board & Carton Co., Inc., New Haven, Conn., a 1948 graduate of Pennsylvania State College.

White, Bruce E., Head, Factory Service, C & S Technical Div., The Buckeye Cellulose Corp., Memphis, Tenn., a 1949 graduate of Louisiana State University.

Wikman, Sten A., Production Director, Papelera Rio Parana S.A., Buenos Aires, Argentina, a 1933 graduate of Harnosands Technical College.

Appointments

James C. Adam, formerly of Beloit Iron Works, is now design engineer for Sandwell International Corp., Portland, Ore.

Gordon L. Adams, formerly of Diamond National Corp., is now Board Mill Coating Supervisor for Interstate Folding Box Co., Miamisburg, Ohio.

John H. Bardsley, formerly of B. C. Forest Products, Ltd., is now Technical Director of Southwest Forest Industries, Snowflake, Ariz.

William C. Bean, formerly of Marathon Corp., is now Technical Director of Columbia River Paper Co., Vancouver, Wash.

Clyde A. Benson is now Technical Superintendent for Garden State Paper Co., Garfield, N. J.

Vernon B. Bodenheimer is now Technical Director of the Continental Can Co., Augusta, Ga.

John J. Bradley, sales engineer for Link Belt Co., has been transferred from Needham, Mass. to Summit, N. J.

Richard K. Britton, chemical engineer for Borden Chemical Co., has been transferred from Bainbridge, N. Y. to Peabody, Mass.

Bengt T. Brunes, formerly of A/B Rosenblads Patenter, is now Vice-President of the Swedish Steam Users Association, Stockholm, Sweden.

Hugh R. Bryson, formerly of Formica, Ltd., is now chemist for Edward Towgood & Co., Samston, England.

John S. Buchanen is now Kraft Mill Superintendent of Nekoosa-Edwards Paper Co., Port Edwards, Wis.

Joseph P. Carey, Jr. is now Head of the Chemical Research Division of Dennison Mfg. Co., Framingham Mass.

Frank L. Cessna, Jr. is now Technical Director of the Sorg Paper Co., Middletown, Ohio.

Harland R. Clodfelter, formerly of Sandwell & Co., is now a consulting engineer with Frankfurter & Associates Seattle, Wash.

George P. Colgan is now Supervisor of Paper Technical Service for Air Reduction Chemical & Carbide Co., Bound Brook, N. J.

Thomas M. Cook, formerly of W. R. Grace & Co., is now Vice-President in Charge of Operations for Penobscot Chemical Fibre Co., Boston, Mass.

Earnest A. Crawford, formerly of W. R. Grace & Co.,

is now a project engineer for the Lummus Co., Newark, N. J.

John R. Dam, formerly of Champion Papers, Inc., is now with the Glidden Co., Baltimore, Md.

Solis Dudnick is now Plant Manager of Sweetheart Cup Corp., Chicago, Ill.

Newell W. Emery, Jr., formerly of Ames Harris Neville Co., is now Chief Chemist of Boise Cascade Kraft Corp., Wallula, Wash.

Eric O. Ericsson is now Assistant Vice-President of Puget Sound Pulp & Timber Co., Bellingham, Wash.

Jacqueline M. Fetsko is now Assistant Research Director of the National Printing Ink Institute, Lehigh University, Bethlehem, Pa.

Frederick R. Gardner, formerly of Hinde & Dauch, Ltd., is now Assistant Laboratory Director of the Hawley-Monk Co., Lockland, Ohio.

Marcel M. Gaschke, formerly of M. W. Kellogg Corp., is now with the Toms River Chemical Corp., Toms River, N. J.

Vincent de P. Gerbereaux is now Director of Marketing Services for Worthington Corp., Harrison, N. J.

John W. Gilbert, of the International Paper Co. has been transferred from Mobile, Ala., to New York, N. Y. as Assistant Director of Research.

Victor Gilpin, formerly of Trinity College, is now Associate Professor of Chemistry, University of Wichita, Wichita, Kan.

Frank H. Glass, formerly of Green Bay Box Co., is now Manufacturing Manager of Mead Containers, Inc., Atlanta, Ga.

F. Claus Globig, formerly of Champion Papers, Inc., is now Supervisor of Papermaking Research and Development for Simpson-Lee Paper Co., Vicksburg, Mich.

Robert P. Green, formerly of Champion Fiber, Inc., is now Assistant Director of Pulp Manufacture Research and Development for Kimberly-Clark Co., Neenah, Wis.

Enrique Gremler, formerly of Black-Clawson Co., is now Technical Assistant to the President of the Upson Co., Lockport, N. Y.

Fritz B. Grunwald is now Works Manager of the South Division of Consolidated Paper Co., Monroe, Mich.

Raymond K. Guinard is now Assistant Manager of New York & Pennsylvania Co., Johnsonburg, Pa.

Kai K. Halsas is now an engineer for Enso-Gutzeit OY, Helsinki, Finland.

John R. Hanzlik, is now Manager of Sales Service for Manchester Machine Co., Middletown, Ohio.

Perry A. Harding, is now Paper Mill Superintendent for the International Paper Co., Chisholm, Me.

Stedman C. Herman is now Adhesives Technical Service Manager for Shawinigan Resins Corp., Springfield, Mass.

Harry F. Highfield, formerly of Noble & Wood Machine Co., is now Technical Director of Steiner Tissue Mills, Albany, N. Y.

Allan C. Hill is now President of the Montmorency Paper Co., New York, N. Y.

George B. Hills, Jr., formerly of Martin Paper Products Division, is now Plant Manager of the Stone Container Corp., Chicago, Ill.

Robert A. Jedeikin, formerly of the American Writing Paper Co., is now President of Jedco Chemical Corp., Mt. Vernon, N. Y.

Donald F. Jenkins, formerly of Champion Papers, Inc., is now Assistant Technical Superintendent of Bowaters Carolina Corp., Catawba, S. C.

Tony Jurecic, formerly of International Paper Co., is now research chemist for Borden Chemical Co., Philadelphia, Pa.

Daniel J. Kuebel, formerly of Champion Papers, Inc., is now with Beveridge Paper Co., Indianapolis, Ind.

Homer L. Kurjan, formerly of C. H. Dexter & Sons, Inc. is now a specialty paper consultant, Windsor, Conn.

William E. Larson is now Acting Production Manager of Champion Papers, Inc. Hamilton, Ohio.

Bjorn J. Lehto, formerly of Allied Paper Corp., is now engineer for Paterson Parchment Paper Co., Bristol, Pa.

Maurice S. Letourneau, formerly with Borden Chemical Co., is now the General Manager of the Chemicals Dept., Wica Chemicals, Charlotte, N. C.

Alberto Levi, formerly of Cartiere Burgo, is now Vice-Director of Cartiera di San Cesario, Modena, Italy.

John E. Maphis, Technical Superintendent of Container Corp. of America, has been transferred from Wabash, Ind. to Los Angeles, Calif.

Vernon L. Marquart, formerly of Radio Corp. of America, is now an engineer with Nuclear Corp. of America, Phoenix, Ariz.

William S. McDonald is now General Superintendent of the International Paper Co., Georgetown, S. C.

Louis A. Melsheimer is now Technical Assistant to the General Manager of the Pigments Division of American Cyanamid Co., New York, N. Y.

Garrett C. Mitchell, Jr. is now Division Manager of New York and Pennsylvania Co., Lock Haven, Pa.

Nello Montagnani, formerly of Cartiere Burgo, is now Technical Director of Cartiera Italiana S.p.A. Torino, Italy.

David E. Peakes, formerly of Northwest Paper Co., is now chemical engineer for Mac Andrews & Forbes Co., Camden, N. J.

Maurice H. Pickard, of U. S. Borax & Chemical Corp. has been transferred from New York, N. Y. to Los Angeles, Calif. as market development and technical service engineer.

Arthur W. Pinkham, Jr. is now Regional Manager of Curtiss-Wright Corp., Princeton, N. J.

Carl E. Price of Becco Chemical Division has been transferred from Buffalo, N. Y. to New York, N. Y. as a technical sales representative.

Robert J. Proctor, formerly with Bauer Bros. Co., is now development engineer for Crocker-Burbank & Co. Assn., Fitchburg, Mass.

Gustaf Ranhagen, formerly with A/B Celleco, is now with Sandwell A/B, Stockholm, Sweden.

James W. Replogle, Jr., formerly with Panellit Service Corp., is now a Shift Superintendent for Rayonier, Inc., Jesup, Ga.

Carleton H. Root is now Director of Research and Development for the Corrugated Container Division of Continental Can Co., Chicago, Ill.

Richard Lee Schmalz is now a patent agent for the West Virginia Pulp & Paper Co., Bethesda, Md.

Rudolf E. Schmut, research chemist of West Virginia Pulp & Paper Co., has been transferred from Charleston, S. C. to Mechanicville, N. Y.

Robert W. Shaw, formerly of Watervliet Paper Co., is now a technical service representative for J. M. Huber Corp., Huber, Ga.

Charles W. Spalding, formerly student at The Institute of Paper Chemistry, is now a research associate with Hammermill Paper Co., Erie, Pa.

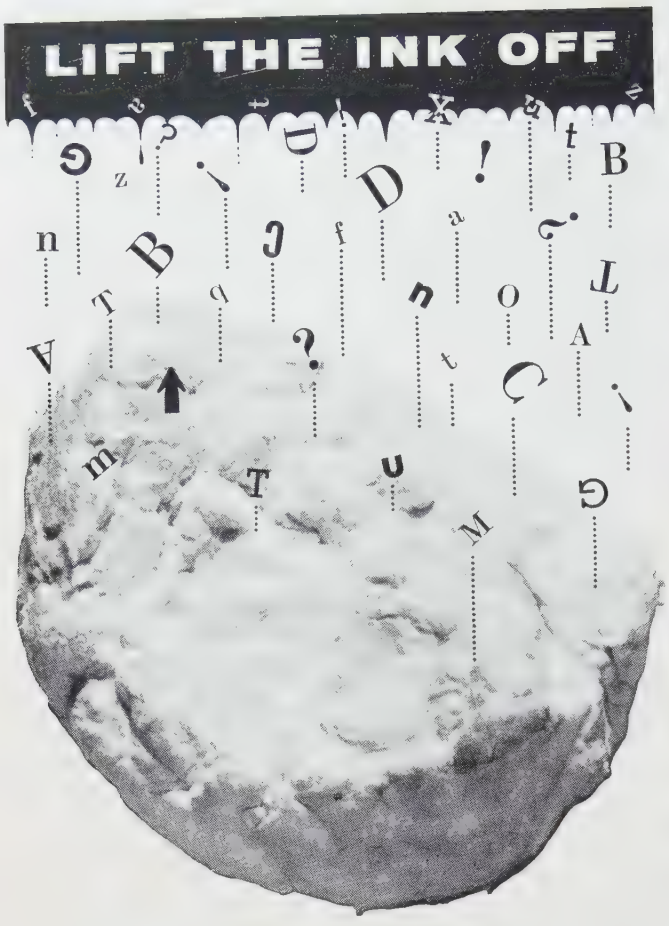
Vivian T. Stannet, formerly Professor of Polymer Chemistry, is now Associate Director of the Camille Dreyfus Laboratory of the Research Triangle Institute, Durham, N. C.

Robert O. Stephenson is now Assistant Division Manager for Champion Papers, Inc., Hamilton, Ohio.

William R. Streed, formerly of Machine Products Co., Inc., is now a systems designer for Atomedic Research Center, Montgomery, Ala.

Mahesh C. Varshney, formerly of Consolidated Water

De-inking?



with **PQ SILICATE**

"Cleaner, whiter" . . . the difference you see in pulp when you de-ink with PQ Silicate. Unlike other alkalies which dissolve the ink, PQ Silicate's superior detergency *lifts* ink from the paper. (Dissolved inks tend to "bleed" back into the stock and thus discolor it.)

PQ Silicates' soluble silica content is effective in preventing the removed ink and soil from re-depositing. Another advantage is that silicate does not yellow ground wood stock. Brightness may be increased as much as five points.

Send for a copy of bulletin 6-1 *"PQ Soluble Silicates for the Pulp & Paper Industry"* describing PQ Silicate Products and their uses in the paper industry.

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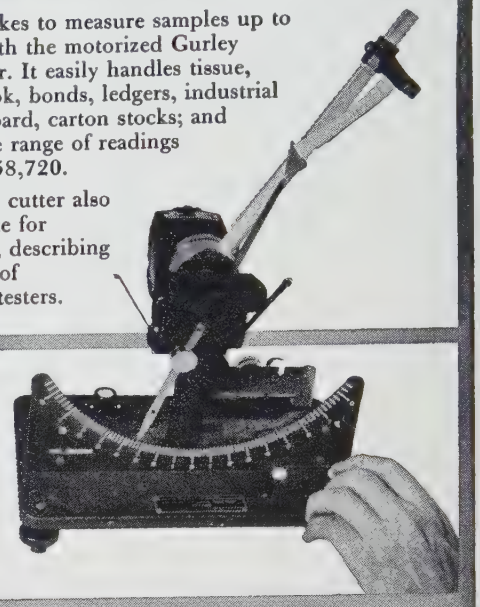
21 SECONDS *to Test Stiffness of Paper and Paper Board*

That's all it takes to measure samples up to 0.25" thick with the motorized Gurley Stiffness Tester. It easily handles tissue, newsprint, book, bonds, ledgers, industrial papers, cardboard, carton stocks; and permits a wide range of readings from 1 to 3,358,720.

Precision strip cutter also available. Write for Bulletin 1400, describing complete line of Gurley paper testers.



W. & L. E. Gurley
Station Plaza & Fulton Sts., Troy N. Y.

Power & Paper Co., is now with Rohtas Industries, Ltd., Dalmianagar, India.

O. James Walker, formerly of Anglo Canadian Pulp & Paper Mills, is now chemist for Consolidated Paper Corp., Grand'Mere, P. Q., Canada.

William R. Walsh, Jr., formerly of Hammermill Paper Co., is now a technical service man with Gilman Paper Co., New York, N. Y.

W. Dale Wegrich is now Manager of Technical Service for Diamond Alkali Co., Painesville, Ohio.

Walter L. Welch of E. F. Houghton & Co. has been transferred from Philadelphia, Pa. to North Leominster, Mass. as New England sales representative.

James H. Wing, formerly of Continental Can Co., is now a project engineer for Standard Packaging Corp., New York, N. Y.

J. A. Van den Akker of The Institute of Paper Chemistry is now a Fullbright Exchange Professor at the Manchester College of Science and Technology in England for the next six months where he will lecture to graduate and undergraduate students in the Paper Section of the college.

Anthony A. Yankowski, formerly of Kimberly-Clark Corp., is now Mill Manager of Irving Pulp & Paper Co., Lancaster, N. B.

Donald M. Zoller, formerly of Champion Papers, Inc., is now a chemical engineer with Diamond National Corp., Middletown, Ohio.

* * *

Louis Camancho, Chief Engineer has succeeded **Sergio Caraveo** as the corporate representative of Cellulosa di Chihuahua, Mexico in the Technical Association.

* * *

F. L. Meacham, President has succeeded **F. R. Henry** as the official representative of Simonds Worden White Co., Dayton, Ohio, in TAPPI.

INDUSTRY NOTES

Pulp and Paper Manufacturers and Converters

Bowaters Southern Paper Corp.

A 650-acre lake, a scenic waterfall, and 80 acres of adjacent woodlands were presented to the State of Georgia recently by Bowaters Southern Paper Corp., Calhoun, Tenn.

Governor Ernest Vandiver accepted the deeds for High Falls Lake, the falls and the land, which are all located some 6 miles south of here, from V. J. Sutton, vice-president and general manager of the Tennessee newsprint mill.

Fulton Lovell, director of the State Game and Fish Commission, then briefly outlined the state's plans for developing the area for recreational use.

Several hundred guests attended the impressive ceremonies beside the lake and falls, and heard Governor Vandiver describe the gift by Bowaters as being "indicative of its desire to serve the community in which it is located and the people who live here—I can think of no greater gift of good citizenship than the one being witnessed here today."

Earlier, Mr. Sutton told the gathering: "Bowaters Southern Paper Corp. and its woods organization, Hiwassee Land Co., are pleased today to team with the government of Georgia to make possible the long-range development of this lake and park area for all the people of this state and nation.

"We are presenting this lake and park to the Georgia Game and Fish Commission because they can assure the greatest public benefit from this property for generations to come."

In noting the direct economic contributions of his company to Georgia, Mr. Sutton said that in 1960 alone Bowaters had paid more than $2,500,000 for pulpwood harvested from privately owned land in Georgia. And, since startup of the mill at Calhoun in mid-1954 through last year, the company paid Georgia timber growers some $13,000,000.

One of the most scenic spots in west central Georgia, near Jackson, High Falls Lake is surrounded by tall trees and offers ideal camping and picnicking areas. Created by an old hydroelectric dam across the Towaliga River, the lake covers approximately 650 acres. The lake and 80 acres of adjacent land were presented by Bowaters Southern Paper Corp., Calhoun, Tenn., to the State of Georgia for use as a public recreational area, in ceremonies held July 25 at the lake

Long popular with fishing, boating, and swimming enthusiasts, High Falls Lake is located in rolling, heavily wooded country about 15 miles southeast of Griffin, and approximately 6 miles southwest of Jackson.

The dam across the Towaliga River, which created the Lake, was built by the old Towaliga Power Co. Organized in 1904, the company operated a 5000-hp. hydroelectric generating plant.

The Hgih Falls plant was acquired by the Georgia Electric Co. in 1925. Then, in May of 1930, control of Georgia Electric was taken over by the Georgia Power Co. which continued to use the plant for a number of years until its operation was no longer economically feasible. The lake property and surrounding timber lands were sold by Georgia Power to the Bowaters Organization in September, 1960.

Bowaters Southern Paper Corp. opened its newsprint mill at Calhoun, Tenn., in mid 1954. The largest newsprint mill in the United States, it now has an annual capacity of 430,000 tons of newsprint and uses more than 500,000 cords of pulpwood annually.

Hiwassee Land Co. conducts its forestry operations in more than 50 north and middle Georgia counties. The company maintains 18 pulpwood railyards in Georgia, and an estimated 2500 independent pulpwood producers and farmers, plus their help, cut and haul wood to the Bowaters landings each year.

Since 1952 Hiwassee has planted 33,000,000 pine seedlings in Georgia. The reforestation program provides seasonal employment to some 200 Georgians.

Champion Papers, Inc.

Champion Papers, one of the world's leading manufacturers of fine publishing, packaging, business, and converting papers and a major distributor of all paper products, has completed a series of changes that gives the 68-year-old company a new name, a new headquarters, and a new set of corporate identity symbols.

Since 1935, when The Champion Coated Paper Co. and The Champion Fibre Co. combined, the company has been known as The Champion Paper and Fibre Co. In recent years, according to Champion's general sales manager, Sterling Brown, "this name has become less and less descriptive as we have moved into both the manufacture and distribution of many new paper and allied products." At the company's annual meeting, therefore, shareholders authorized changing the name to Champion Papers, Inc.

At the same time, the company's corporate staff and executive offices have moved from their nearly forty-year-old quarters in Hamilton, Ohio, birthplace of the parent company, into a just-completed, modern office building, also in Hamilton, which is regarded as one of the nation's most architecturally advanced and efficient business structures. The new glass and stone building enables the economical drawing together of offices which, in the past few years, have been scattered over a dozen separate locations in the company's home town. Address of the new building, reflecting the company's traditional trademark symbol of a mounted knight-champion, will be Knightsbridge, Hamilton, Ohio.

Coosa River Newsprint Co.

P. A. Bachelder, president of Coosa River Newsprint Co., of Coosa Pines, Ala., has announced that a joint study group has been formed to consider the possible applications in the

paper industry of process controls by computers. This study will be conducted in the Alabama area, using personnel, facilities, and equipment of Coosa River Newsprint Co., International Business Machines Co., and The Rust Engineering Co.

The joint effort is one of mutual interest by three firms prominent in their respective fields. IBM is one of the leaders in the data processing and computer field; Coosa River Newsprint Co. is one of the leading producers of newsprint, and The Rust Engineering Co. has long been prominent as a service company performing engineering and construction work for the paper industry.

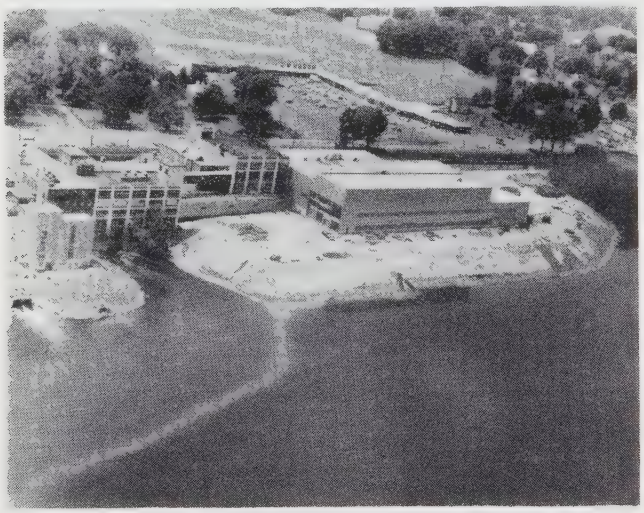
Crown Zellerbach Corp.

Two appointments to management posts in Crown Zellerbach Corporation's manufacturing division were announced recently by F. O. Boylon, vice president: **Leland A. Kelson** has been appointed resident manager of the Lebanon, Ore., mill division; and **Clarence E. Englund** has been named assistant resident manager at the West Linn, Ore., mill division.

C. H. Dexter & Sons

C. H. Dexter & Sons, Inc., has a $5 million plant expansion now under way. The new facilities, including a power plant, a specially designed paper machine and supporting equipment, rewinding department and pulp mill, are scheduled for completion next year.

C. H. Dexter & Sons, Inc., is a 194-year-old firm specializing in the manufacture of "specialty" papers and webs. The



Aerial view of C. H. Dexter & Sons' new plant expansion in Windsor Locks, Conn. New power plant is at far left, new plant at right. The manufacturer of specialty papers has occupied the same site on the Connecticut River since 1767

firm is the originator and leading supplier of tea bag paper, meat casings and stencil paper, produces air and oil filter papers, and is pioneering the development of micro-glass, micro-quartz, and synthetic fiber webs.

The current program is the third phase in the expansion and modernization of Dexter's manufacturing and research facilities. The first two phases, completed in 1958, resulted in the installation of two new high-speed specialty paper machines of Dexter's own design, together with supporting equipment.

Fitchburg Paper Co.

A pioneering New England paper company with almost a century of tradition behind it is making plans to use atomic power in its production of paper.

Fitchburg Paper Co., of Fitchburg, Mass., whose corporate roots extend back to the kerosene lamp days of 1864, has applied to the Atomic Energy Commission to participate in a demonstration low-temperature process steam generating project incorporating a nuclear reactor. A low-temperature process heat reactor is one designed to fit the needs of industrial operations such as those in paper manufacturing, where large amounts of heat are required.

According to AEC plans, the reactor project should be ready for operation in about three and a half years. The design would be based upon existing technology, and have an output capacity of from 30,000 to 40,000 thermal kilowatts at steam pressures ranging from 15 to 200 p.s.i.

The Fitchburg Paper Co. is one of the nation's leading manufacturers, developers and converters of technical and specialty grades of paper for the printing, packaging, plastic laminating, photo, office copying and reproduction industries.

Georgia-Pacific

Stock Purchase Through Payroll Deduction

Approximately 9500 hourly employees scattered over 32 states received a brief questionnaire at their homes, asking whether they would or would not like to be able to buy G-P stock by having a certain amount deducted from their pay check each month. Better than 87% said they would. A place on the questionnaire for remarks brought almost unanimously favorable comments, most of them talking about mutual interest in the company, stock as a form of saving, and the general stimulation such a plan would provide.

Company officials said that, as a result of this substantial interest, they are studying various ways to put a stock-purchase program into effect. Indirectly a number of G-P employees participate in company progress through union pension funds, a substantial part of which is invested in Georgia-Pacific stock.

Central Research Laboratory

Expansion of the research staff at its Central Research Laboratory in Beaverton, Ore., as part of its research and development program has been announced by Georgia-Pacific Corp. Robert W. Hess, research director, said that two specialists, one a chemical engineer and the other a research engineer, have been selected. They are **H. John Ballo** and **Roland A. Haertl,** respectively.

White Containers, Inc.

White Containers, Inc., of Monticello, Iowa, has been acquired for an undisclosed sum and will become a part of the Georgia-Pacific Paper Co. container group, operating under the G-P name. White has a capacity of 10 million sq. ft. of corrugated production per month, produce in a 40,000 sq-ft. plant. The Monticello plant, located 25 miles southwest of Dubuque, serves customers within a 250-mile radius. The new owners anticipate no personnel or management changes. The operation employs 35 persons.

International Paper Co.

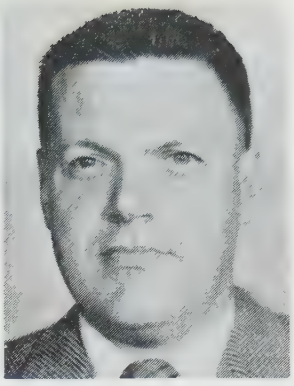
Appointments

James B. Malloy has been appointed resident manager of IPC's new Container Division plant near Pittsburgh, Pa.

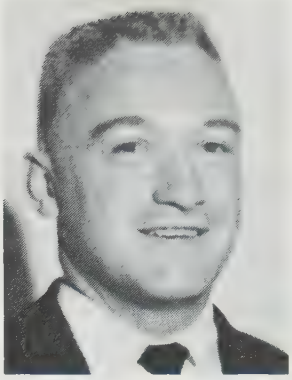
The new Container Division plant is being built on a 20-acre tract near the New Stanton interchange of the Pennsylvania Turnpike on new Route 71. The 118,000-sq.-ft. structure is expected to be in operation early next spring. It will employ approximately 150 persons and will serve corrugated shipping container customers in the tristate area of Pennsylvania, Ohio, and West Virginia.

John W. Gilbert, Jr., has been named assistant director of research and **John A. Lyden,** assistant to the vice-president in charge of manufacturing of International Paper Co. Both men will make their headquarters in New York City.

Hubert F. Parker, New York & Pennsylvania Co.



Garrett C. Mitchell, New York & Pennsylvania Co.



Samuel A. Wood, New York & Pennsylvania Co.

Allen B. Campbell, New York & Pennsylvania Co.

THE MEAD CORP.

Forestry Research Pact

The Mead Corp. and the U. S. Forest Service signed an agreement to jointly conduct research in the management of hardwood timber for the production of pulpwood.

This new research program will be conducted on the 700-acre Mead Experimental Forest located in southwestern Ohio. (This is five miles northwest of Lucasville and adjacent to the Molly Lauman Girl Scout Camp.)

Answers will be sought to such questions as: (1) How and when do you harvest pulpwood trees so as to assure good forest stand for the future? (2) What steps can be taken to increase the growth rate of pulpwood as well as sawtimber trees? (3) What is the optimum age for harvesting a pulpwood tree?

NEKOOSA-EDWARDS PAPER CO.

John S. Buchanan has been named superintendent of the Nekoosa kraft mill of Nekoosa-Edwards Paper Co. He served as acting superintendent since January 1 of this year, and prior to that was technical assistant to the kraft mill superintendent.

NEW YORK & PENNSYLVANIA CO.

The company has announced a series of advancements following the retirement of **Hubert F. Parker,** division engineer of the Lock Haven operation.

Garrett C. Mitchell moved up to the top post of division engineer. His former position, maintenance superintendent for the division, was filled by **Samuel A. Wood.** Another assistant to Mr. Mitchell, **Allen B. Campbell,** is now electrical maintenance supervisor.

The company has also announced the realignment of the management at its Johnsonburg, Pa., paper mill. Newly appointed manager of the Johnsonburg Division is **Henry H. Bell,** formerly general superintendent. He succeeds **R. Neal Jones** who retired this month after 34 years in that position.

Assuming the post of assistant manager is **Raymond K. Guinard,** who had been technical director. New general superintendent is **Francis W. Crowe,** previously assistant general superintendent.

OWENS-ILLINOIS GLASS CO.

The consolidation of all its Forest Products Group divisions and subsidiaries into one operating division and the creation of five regions to operate the company's 21 corrugated shipping box plants has been announced by Owens-Illinois Glass Co.

The selection of **Edwin D. Dodd,** an Owens-Illinois vice-president and now general manager of the Paper Products Division, as general manager of the new Forest Products Division was announced at the same time by Hugh C. Laughlin, Owens-Illinois executive vice-president in charge of forest products operations.

The new division will combine the three divisions and one major subsidiary which now make up the Forest Products Group—the mill, multiwall bag, and paper products divisions and National Container Corp. of California.

PACKAGING CORPORATION OF AMERICA

Expandable Plastics Co.

Packaging Corporation of America, of Evanston, Ill., has acquired Expandable Plastics Co., of Akron, Ohio, as part of its continuing expansion in the field of expanded polystyrene—foam plastics—packaging. The Akron plant is the fourth plastics operation to be acquired or launched by Packaging Corp. since it entered the foam plastics field last December. The company now is the nation's largest custom molder of expanded polystyrene plastics.

H. H. Bell, New York & Pennsylvania Co.

R. Neal Jones, New York & Pennsylvania Co.

R. K. Guinard, New York & Pennsylvania Co.

F. W. Crowe, New York & Pennsylvania Co.

Penobscot Chemical Fibre Co.

Eugene H. Clapp, president of Penobscot Chemical Fibre Co., announces that **Thomas M. Cook,** vice-president of the W. R. Grace Co., Paper Division, became vice-president in charge of manufacturing and operations of the company on July 17, 1961.

Price Bros. & Co.

Price Brothers & Co., Ltd., of Quebec City, Canada, recently awarded a contract to the Black-Clawson Co. for construction of a new 156-in. paper machine. The new machine is being built in Canada by Black-Clawson (Canada) Ltd. and will produce 100 tons of 40-lb. kraft per day. It will also make grades ranging from 25-lb. paper to 0.009 and 0.016 board, as well as newsprint. The new machine is scheduled for installation in the Price Brothers' mill at Kenogami, Que.

Scott Paper Co.

Scott Paper Co.'s board of directors has elected **Charles D. Dickey, Jr.,** a vice-president of the corporation. In a newly created position, he will have executive responsibility for treasury and corporate purchasing functions and will report to the administrative vice-president, who is one of Scott's three-man executive committee.

Mr. Dickey has been on loan from Scott, since 1957, to British Columbia Forest Products Ltd., where he served as president.

St. Regis Paper Co.

St. Regis Paper Co. announces that **Donald O. Sensenbach** has been appointed general manager of the Engineering and Machine Division, effective Sept. 1, 1961. Mr. Sensenbach succeeds **R. McKim Browning** who has resigned from the company. Mr. Sensenbach will be located at the divisional headquarters in Providence, R. I.

Standard Packaging Corp.

Standard Packaging Corp. has acquired the business of the Golden Fleece Tissue Mills, Inc., and its wholly owned subsidiary Battenville Paper Mills, Inc. Harry Preston, founder of the company, Stanley Preston, and Sherman Wiesen, the present Executive Management group, will continue in active operation of the business as a division of Standard Packaging and it will become a part of the Consumer Products Group of Standard Packaging.

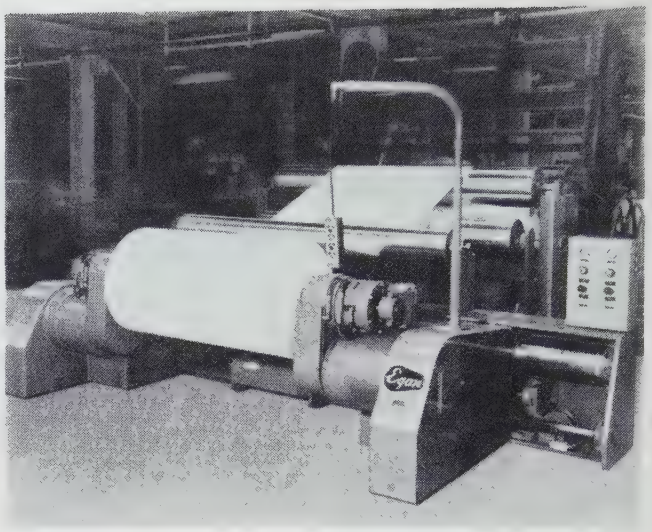
Golden Fleece has as its principal business the making, converting, and packaging of high-quality facial tissue at its plants located in Battenville and Greenwich, N. Y. The majority of Golden Fleece's sales consists of boxed facial tissues sold under the private label of well-known retail chains, supermarkets, and drugstores.

S. D. Warren Co.

New Type Shaftless Unwind Installed

The first of Frank W. Egan & Co.'s new shaftless unwind stands has been installed in the S. D. Warren plant, Cumberland Mills, Me. The unit operates at speeds up to 1000 f.p.m. It will handle rolls 84 in. wide with diameters up to 50 in.

New design features of the Egan shaftless unwind have resulted in production of a more compact unit. This has been accomplished by eliminating the need for a cross-shaft between the pickup arms. Constructed for floor pickups, the shaftless unwind is easily handled by one man. Motorized chucking of cores with 3-in. inside diameter and up as well as skew adjustment are standard features. Automatic sideshifting and constant tension devices are optional items.



Egan's shaftless unwind stand which has been installed in S. D. Warren Co. plant, Cumberland Mills, Me.

Weyerhaeuser Co.

Research

Completion of expanded facilities for pulp, paperboard, and container research was announced recently by J. D. Reagh, director of Weyerhaeuser Co.'s Longview, Wash., pulp research center. Expansion of laboratory space, additions to the research staff, and extensive new equipment are directed especially to meeting customer and market requirements with Weyerhaeuser paperboard and container products.

A new two-story wing has been added to the pulp research building, increasing space approximately 50%. This provides for the installation of a new pilot coating machine, its related operating equipment, complete hot-dry and cold-moist environments for testing existing products in market application and new products under development.

With installation of the new pilot coating machine, a program of experiments and investigations of coating formulations, equipment and conditions will be initiated under the direction of Gary W. Jones, head of the paperboard and coatings research group. Similarly, the new container board and container test facilities will enlarge the scope of research on customer problems and on programs initiated from within the company, under the direction of R. M. Morris, head of the container board research group.

Staff appointments announced by Dr. Reagh are **Patrick Trotter**, from The Institute of Paper Chemistry, Appleton, Wis., and **Myron Marander**, from the Fiberboard corporation, Antioch, Calif.

The expanded research facilities and added staff will provide Weyerhaeuser's existing and planned production plants in Plymouth, N. C., and Longview, Wash., with pilot run guides and new strength in production development. The objectives of the department's research will be to improve container board, paperboard and coating formulations and processes, quality control methods and tests, and continuing tests for paperboard and container board characteristics that meet the specialized needs of the market.

Educational Institutions

Arizona State University

"How to Obtain Valid Data on Purpose" is the theme for the new short courses on Measurement Engineering to be offered at Arizona State University, Jan. 29 to Feb. 2, 1962. It is the purpose of these short courses to present the Science of measurement in a logical and orderly manner. The short courses consist of a lecture program and a concurrent experi-

mental program. About one half of the lectures concentrate on the basic physical principles on which transducers are based and on fundamental measurement theory including static and dynamic system behavior and the correlation between them. Particular emphasis is placed on the interaction between system components and on how measurement systems are properly planned and designed. The other half of the program is devoted to measurement problems and approached in specific areas.

The speakers have been selected for their complete familiarity with both the theoretical and practical aspects of their special fields, and for the contributions which they have made to advance the state of their own art.

This series of lectures is prepared for the dual purpose of presenting a review of some of the advanced techniques, while at the same time including elementary and fundamental information necessary for the novice who wishes to commence work with measuring systems. Each registrant will be provided with text and notes covering the fundamentals.

The program is directed toward laboratory workers, supervisors, designers, theoretical analysts, and others who may be interested in obtaining an insight into the operation of measuring devices and the engineering design principles associated with the systems. The academic level is suitable for those with a Bachelor's (or higher) degree in engineering, physics, or metallurgy; however, in certain cases, experienced technicians without degrees can be expected to gain full benefits from the programs.

Although the lectures have been prepared largely from the point of view of the nonelectrical engineer, the topics to be discussed have been selected to provide maximum benefit for those who are already skilled in measurements but who possess little experience in electrical instrumentation as such, and for the electronics expert, who may not be so familiar with mechanical systems.

Registration lists close on Jan. 19, 1962. Contact Prof. Peter K. Stein, Engineering Center, Arizona State University, Tempe, Ariz., the Director for the Short Courses, for further information. Fees for the programs are $200 for the lecture program and $125 for the experimental program.

Syracuse

American Cyanamid Research Grant

Frank W. Lorey, associate professor of pulp and paper technology and pilot plant group leader, was recently awarded a research grant by The American Cyanamid Co. of Stamford, Conn.

The grant, amounting to $3000, is to be used for a study to evaluate selective papermaking chemicals.

The Institute of Paper Chemistry

New Electronic Computer System Installed

The installation is among the first of its kind in the nation. It consists of the IBM 1620 data processing system with the 1622 buffered card reader-punch. It works 16 times faster than the 610 system previously used, and will accept "algebraic language."

The system is being placed at The Institute under the nationwide "University Program" of the International Business Machine Corp., in which part of the rental charge is contributed by IBM.

In addition to scientific research uses, the new system will be used in a machine computation course which the Institute has offered for several years. Larger and more complicated problems encountered in this course and in student research, which were previously run on large computer systems outside the Institute, will now be handled internally on the new equipment.

Dr. Edward F. Thode, chairman of the Institute's computer committee, said the new system's buffered card reader-punch has its own "memory," which allows it to read and store up to 80 alphabetical or numerical designations punched on a card. This allows the computer portion of the system to keep on working even while information is being fed into the overall system. In 3.6 microseconds, the buffered card portion can transfer its information to the computer portion of the system.

An IBM 1620 data processing system gets its initial run at The Institute of Paper Chemistry at Appleton, Wis. James C. Feese, IBM representative, points to lights indicating the computer's operation on the machine's console. Dr. Edward F. Thode, standing, chairman of the Institute's computer committee, and John Bachhuber, seated, supervisor of machine computation at the Institute, watch the operation

According to Dr. Thode, specific uses of computer systems at The Institute were in the mathematical calculation of statistics, solution of complex equations in engineering and physics, and processing of large amounts of data in scientific problems. The new system will also be used as a mathematical tool in developing new techniques for solving quantitative problems.

Indian Paper Productivity Team

Ten men from India touring the United States met two of their countrymen at The Institute of Paper Chemistry on August 15. A paper productivity team from India's pulp and paper industry spent the day at The Institute of Paper Chemistry as part of their study in the United States of new developments in papermaking and techniques of paper production.

In several sessions with the Institute staff and in the Institute's research laboratories, the ten Indians looked particularly into cellulose chemistry and dissolving pulp, pulping, and recovery practice. Also interested in containers, they viewed the extensive testing and experimental facilities of the Institute's container laboratory.

The two countrymen they found waiting for them were M. G. Karnik and A. V. Iyer. Karnik is a visiting scientist at The Institute of Paper Chemistry, conducting special research under Dr. Kyle Ward, Jr., chief of the Institute's cellulose group. Karnik is on the staff of the cellulose and paper branch of the Forest Research Institute at Dehra Dun (Darra Doon), India. Iyer is on a post doctoral fellowship conducting research in the Institute's physics group. He is an instructor in electrical engineering at Birla (Burla) Engineering College, Pilani (Puh la/ nee), Rajasthan (Rah juh stan/), India.

The Indian Paper Industry Productivity Team's tour of the United States was under the auspices of the International Co-operation Administration, a branch of the U. S. State Department.

National Printing Ink Research Institute, Lehigh University

William D. Schaeffer has been promoted to associate research director and **Miss Jacqueline M. Fetsko** to assistant research director. They have lectured widely in the United States and abroad, and are members of TAPPI, the Technical Association of the Graphic Arts, and Sigma Xi, national research honorary. Mr. Schaeffer is also a member of the New York Academy of Science and the American Chemical Society. Miss Fetsko is active in the American Contract Bridge League, of which she is a Life Master, the American Association of University Women, and Kappa Delta Sorority.

Packaging School at Manchester, England

The very successful completion of this school held at the College of Science and Technology in Manchester, England, marked an important development in the packaging industry in that country. It was the first of its kind to be arranged by the College and by The Printing, Packaging and Allied Trades Research Association, and the first series of lectures in packaging other than those arranged by the Institute of Packaging.

The organization was in the hands of the Paper Section of the Textile Chemistry Department of the College and The Printing, Packaging and Allied Trades Research Association and the Institute of Packaging together. Almost every aspect of the use of paper and board alone or in combination with other materials was covered, together with principles of design and testing of paper and board packages. Visits to industrial firms making and using packages were included in the Summer School program. The evenings were largely spent in social activities where members attending the school could meet and discuss matters of common interest informally.

There have already been a number of requests for copies of the lecture notes issued to the members of the school, and the organizers are considering publishing these as a monograph. It would help to assess demand if anyone who is likely to be interested in obtaining copies would send his request to the Registrar of the Manchester College of Science and Technology, Manchester, 1.

University of Maine

Paper Technology Session

Planning, leadership, and good communications are paramount to successful paper mill management, according to statements by a panel of three top paper mill executives at the final session of the summer pulp and paper institute program here.

For the better part of four hours the 67 students enrolled in the institute questioned John H. Heuer, Bangor, vice-president of the Great Northern Paper Co., George Olmsted, Jr., Boston, president of S. D. Warren Co.; and Ralph A. Wilkins, East Walpole, Mass., president of Bird & Son, Inc. Austin H. Peck, vice-president for academic affairs of the University of Maine, was chairman of the panel.

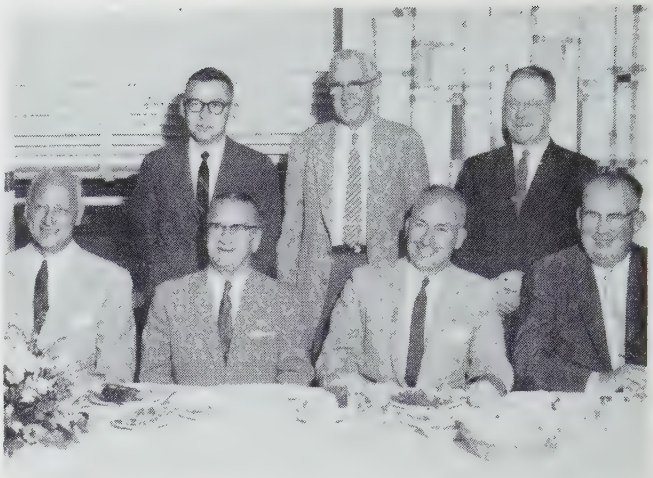
Training technical manpower was considered as one of the key problems of the industry. The discussion included qualifications of managers, company organization, the principles and functions of management, labor relations, and production and control.

The session was the last one of two 3-week programs, the first of which was devoted to pulp technology, and the second to paper technology.

Professor Lyle C. Jenness, head of the department of chemical engineering at the University, was the director of the Institute.

Pulp and Paper Foundation Officers

J. Larcom Ober of Salem, Mass., vice-president (retired) of Scott Paper Co., was elected chairman of the board, and **Frederic A. Soderberg,** vice-president of Huyck Corp., Rensselaer, N. Y., was chosen president of the University of Maine Pulp and Paper Foundation at the annual meeting of that organization.



Head table at the Paper Technology program graduation Aug. 17, 1961—Second Summer Pulp and Paper Institute. Left to right: Seated—Frederick A. Soderberg, Professor Lyle C. Jenness, Dr. Lloyd H. Elliott, and John H. Heuer. Standing—Dr. Austin H. Peck, Charles E. Crossland, and Dean Weston S. Evans

Manuel C. McDonald of Bangor, president of Great Northern Paper Co., and **Ralph H. Cutting** of Waterville, president of Keyes Fibre Co., were named vice-presidents. **Ralph A. Wilkins** of East Walpole, Mass., president of Bird & Son, Inc., and **Henry W. Fales,** vice-president of St. Croix Paper Co. of Woodland, were re-elected treasurer and secretary of the nonprofit corporation.

Lewis Calder, Jr., of New York City, executive vice-president of Kennebec Pulp and Paper Co.; **Eugene H. Clapp** of Boston, Mass., president of Penobscot Chemical Fibre Co.; **Alvah Crocker** of Fitchburg, Mass., president of Crocker Burbank & Co.; **Harry C. Moore** of Beloit, Wis., president of the Beloit Iron Works; and **David W. Stapleton** of Newton Upper Falls, president of Stowe-Woodward, Inc., were elected directors at large for three-year terms.

Ex-officio directors are **William H. Chisholm** of New York City, president of Oxford Paper Co., and a trustee of the University of Maine; **Dr. Lloyd H. Elliott,** president of the University of Maine; and **Dean Weston S. Evans,** dean of the College of Technology at the University.

Yeshiva University

Joseph M. Mazer, treasurer of the Hudson Pulp and Paper Corp., New York City, and an outstanding Jewish communal leader, has been named chairman of Yeshiva University's year-long 75th anniversary celebration which began in September.

Mr. Mazer, who is a national chairman of the United Jewish Appeal and the American Friends of the Hebrew University, and deputy chairman of that University's Board of Governors, is a director of the Jewish National Fund and the Palestine Foundation Fund as well as one of the leaders of the State of Israel Bond drive.

He will head the Diamond Jubilee Anniversary Committee, of which President John F. Kennedy is honorary chairman, which will plan and coordinate all of the Diamond Jubilee Year events.

Following in the footsteps of his distinguished father, the late Abraham Mazer, in every important communal and philanthropic endeavor, Mr. Mazer succeeded him as a member of the Board of Trustees of Yeshiva University in 1950.

Industry Suppliers

ALLIED CHEMICAL CORP.

Appointments

The appointment of six regional sales office managers and five assistant managers for Allied Chemical's General Chemical Division has been announced by Frank J. Woods, director of sales.

The new sales managers and their offices are: **Arthur H. Baker,** New York Metropolitan; **Albert B. Connelly,** Houston; **Harold E. Donaldson,** St. Louis; **William P. Doyle,** Buffalo; **Edmund R. Lett,** Birmingham; and **Wesley G. Webster,** Denver.

The new assistant sales managers and their offices are: **Lester I. Adams,** New York Metropolitan; **Joseph M. Byouk,** Chicago; **Stephen J. Muller,** Los Angeles; **Richard J. Regan,** Philadelphia; and **James E. Strader,** Chicago.

THE LOUIS ALLIS CO.

Bulletin on Digital Process Controllers

The Dynapar Corp., subsidiary of The Louis Allis Co., has recently published Bulletin No. 201-A on Digital Process Controllers for industrial counting, measuring, indicating and control applications.

Designed for high speed, high accuracy industrial requirements, Dynapar digital process controllers accurately count pulses from all types of sensing devices and control various functions of other equipment when a preset count is reached.

They provide a range of zero to above 180,000 counts per minute with instantaneous reset as standard and optional ranges to 75,000 counts per second with instantaneous reset.

Write The Louis Allis Co., Dept. P, 427 E. Stewart St., Milwaukee 1, Wis.

ALLIS-CHALMERS MFG. CO.

New Close-Coupled Pump Leaflet

Allis-Chalmers compact close-coupled, fractional horsepower, single stage-single suction pump (Model C-15—Type RHK) designed for low-cost handling of clear noncorrosive liquids at temperatures up to 180°F. is described in new literature released by the company.

Available in a range through 48 g.p.m. with heads to 100 ft., the new pump can be installed horizontally or vertically. Casings have centerline discharge, are self-venting and can be rotated to any of four 90° positions.

Copies of the leaflet, 52B1010, which carries performance curves and dimensions, are available on request from Allis-Chalmers, Milwaukee 1, Wis.

Direct-Mounted Motors on Low-Head Screens Help Cut Capital Investment, Maintenance Costs

New literature describes how the direct-mounted motor on the low-head screen saves headroom, reduces belt cost and wear, and simplifies maintenance.

Diagrams illustrate how the direct-mounted motors eliminate overhead superstructures for mounting motors, complicated gantry-type motor supports, and belt tensioning and alignment problems associated with conventional screen motor mountings.

Copies of "The Motor Rides Piggyback." 26B9996, are available on request from Allis-Chalmers, Milwaukee 1, Wis.

Literature Describes New Wedgtite Screen Tensioners

Wedgtite tensioners for cloth and plate deck Allis-Chalmers screens are described in new literature released by the company.

Designed to provide full takeup without changing bolt length, the Wedgtite tensioners eliminate tedious threading and bolt-cutting and reduce downtime. They can be adjusted—$1^1/_4$ in. takeup on each side and $2^1/_2$ in. overall—while the screen is running.

Copies of "New Wedgtite Tensioners," 26B9995, are available on request from Allis-Chalmers, Milwaukee 1, Wis.

Appointments

Leonard J. Linde has been appointed director of engineering, Industries Group, Allis-Chalmers. He had been director of engineering services since August, 1960.

Mr. Linde will be in charge of the overall administration, review and coordination of Industries Group research and development efforts.

L. J. Frick has been appointed to the new position of manager, Industrial Equipment Division sales, Midwest region.

Since 1959, Mr. Frick had been manager, general industrial sales, St. Louis district. He is succeeded in that post by **J. C. Treible.**

AMERICAN POTASH & CHEMICAL CORP.

Appointments

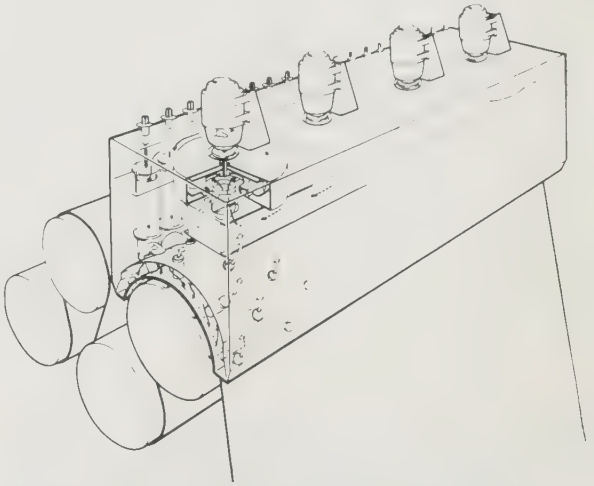
Harry L. Carroll was appointed district sales manager for American Potash on July 1. He assumed responsibility for marketing industrial chemicals in the southeastern area, with headquarters in the company's Atlanta regional sales office.

C. L. Straughan continues as district manager in charge of potash and agricultural chemical sales.

AMERICAN SF PRODUCTS, INC.

Compact SF Air Drier Lessens External Ductwork

A radically new drier for paper, the SF Air Drier, Type C, has been introduced by American SF Products, Inc.,



SF Type C Air Dryer

of Englewood Cliffs, N. J. According to Karl F. Gothner president, the new drier uses the principle of internal air circulation to provide a compact installation on either new or old paper machines within a minimum of area.

The C-drier fits individual cylinders of the papermaking machine.

THE BAUER BROS. CO.

Versatile Crusher-Shredder

Bauer's No. 7-AB crusher-shredder, a heavy duty unit with a wide range of crushing, precrushing or shredding applications in operations where a minimum of fines is desired, is described in Bulletin G-6, now available from The Bauer Bros. Co., 1715 Sheridan Ave., Springfield, Ohio.

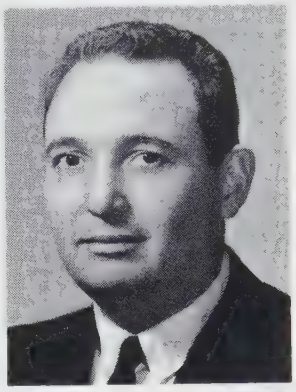
The unit can be used for crushing lumpy chemicals, copra, clay, coke, shells, cobs, and similar materials. When used for shredding such materials as sheets (or webs) of wood pulp,

foam rubber, plastics, and similar materials, the No. 7-AB produces flakelike particles of good uniformity, at high capacity, with very little dust or fines.

BERKSHIRE COLOR & CHEMICAL CO.

Appointment

Berkshire Color, producers of dyestuffs, organic chemical intermediates and auxiliary chemicals, has named **Joseph A. Paciello** manager of its Paper and Leather Division. Mr. Paciello has 22 years of experience in the pulp, paper, and leather industries. He was associated with E. I. du Pont de Nemours & Co. for twenty years and then joined the Amalgamated Leather Co. as assistant to the president.



Joseph A. Paciello, Berkshire Color & Chemical Co.

He is well known for his technical knowledge of the industries and is considered an authority in the application of dyes and chemicals to paper and leather. His record includes service to his field in the following posts: affiliate chairman, PIMA, 1959–1960; member of executive and planning committee, TAPPI, Delaware Valley Section, 1952–1958; secretary and treasurer, Pennsylvania, New Jersey, Delaware Division of PIMA, 1950–1952; member of executive committee, Pennsylvania, New Jersey, Delaware Division, of PIMA, current.

THE BLACK-CLAWSON CO.

New Coating Pilot Plant and Laboratory at Dilts in Operation

In line with its policy of continued emphasis on Research and Development, Black-Clawson has recently completed a major expansion in its facilities with the startup of a second pilot plant and lab at the Dilts Division in Fulton, N. Y.

One of the most modern and complete in the industry, the new facility is primarily designed for customer service work. However, it is also an excellent research tool and is used to develop new products and improve existing products in the extensive Black-Clawson line of coating and converting equipment.

Use of the new commercial size pilot plant and lab is made available to paper and board manufacturers, converters, producers of coating materials and chemicals for studies designed to solve customer problems and develop new products. Typical studies being made include:

Coating Formulations. (a) Development of new coating formulas that will run best in the Flexiblade coater for all types of printing applications. (b) Studying the effects of coating variables, i.e., solids, viscosity, water holding characteristics, etc., on Flexiblade and air doctor operation.

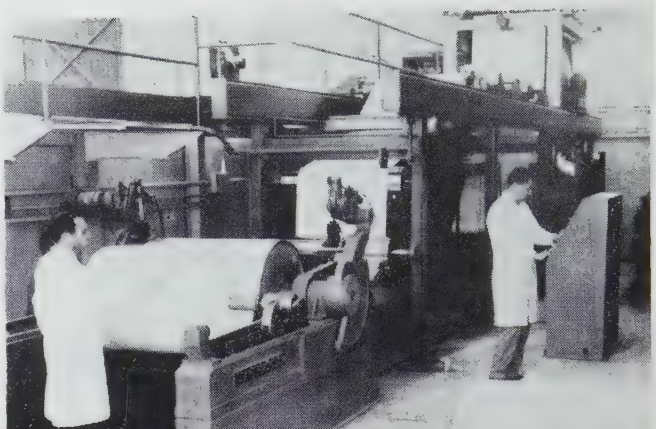
Flexiblade Coating Studies. (a) Study effects of varying blade pressure, box pressure, and coating formulations over complete speed range, 200 to 3000 f.p.m. (b) Comparison of results on various substrates.

Drying studies and studies of double coating—wet on wet.

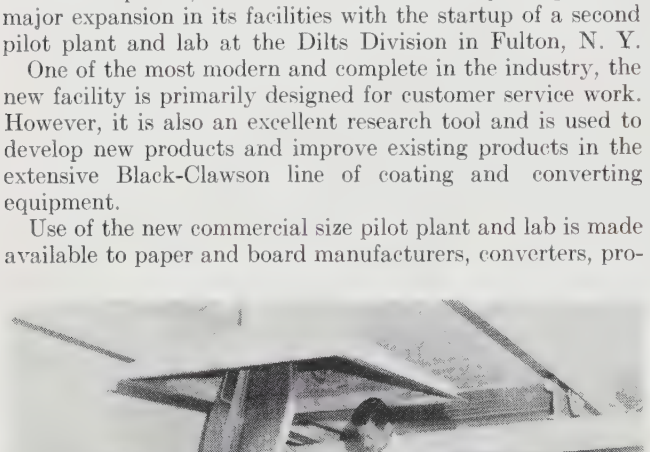
Demonstrations other than coating being made are on the Surfastart 1000 reel, unwind, drive, and color preparation system.

Pilot Plant Equipment Includes Commercial Size Coating Line. A full-size, high-speed clay coating line, complete from unwind to reel has been built by Dilts and installed in the new research area. Designed to do Flexiblade coating, the line will be expanded to increase the lab's versatility as new developments come along. First to be added is an air doctor to follow the Flexiblade for investigation of wet-on-wet studies.

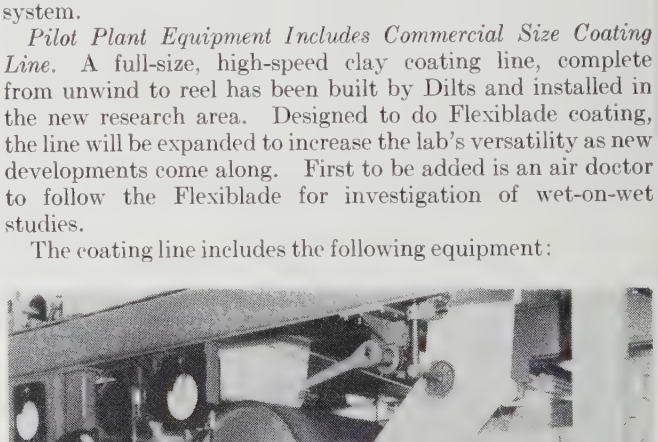
The coating line includes the following equipment:



Pilot clay coating machine line in new Dilts laboratory is full commercial size. Surfastart 1000 winder is in foreground



Loading clay into Kady mill prior to mixing in Black-Clawson's new coating, pilot plant at Fulton, N. Y.



Robert Ingison, technical director (left) and Frank Beckman, research engineer, check equipment during pilot run

Single Position Unwind Stand—handles 60 in. maximum diameter rolls.

Flexiblade Coater—This high-speed coating line embodies the latest design of the commercial Flexiblade Coater.

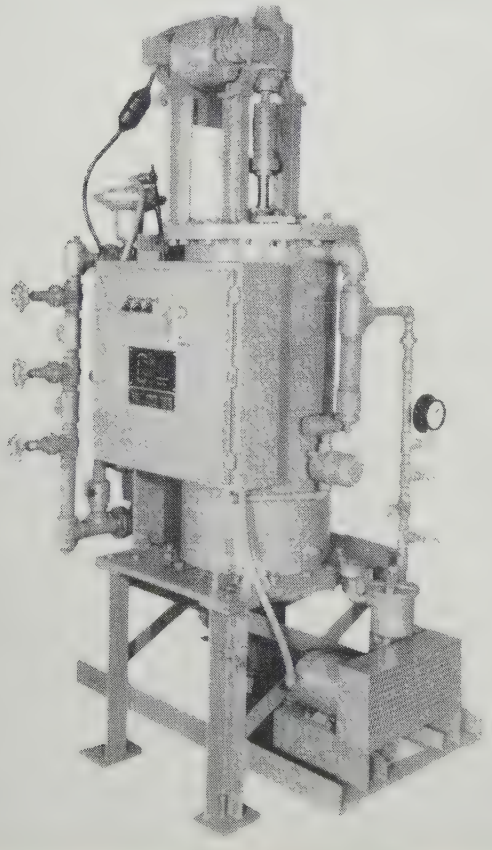
The Flexiblade design of blade coater has several advantages in that coating is delivered vertically to the web from a positively displaced reservoir. This is maintained by positive pumping. The reservoir is deckled for edge control and pressurized for controlling feed. A pressure recorder-controller automatically controls the pressure in the coating chamber. The flexing blades of various calipers are so positioned to give maximum control of light or heavy feeds. Individual adjustments in blade pressure can be made at regular intervals across the entire web to insure uniform coat weight distribution.

Drier Section—The coating line is equipped with a Black-Clawson high velocity 3-D drier manufactured by the Air Systems Division at Hamilton, Ohio, a 60-in. diam. coded steam drum is used in conjunction with the 3-D drier. The drier is gas-heated with a maximum output of 1,250,000 B.t.u. per hr., complete with all the necessary safety controls. The drier is capable of delivering air for drying at velocities up to 18,000 f.p.m. through cross slots located $^1/_2$ in. from the web.

Surfastart 1000 Winder—The surface winder consists of a 30-in. diam. tubular steel drum mounted in antifriction bearings. A pair of air loaded, pivot type arms support a shaft and chuck arrangement for the cores. The pneumatic loading arrangement allows the operator to adjust the pressure setting on the arms to control the hardness of the finished roll.

The primary arms transfer the winding roll to the horizontal slide ways allowing make ready with new core for roll change.

Addition of the centerwind attachment allows the operator to do either surface winding alone, surface winding with centerwind assist, or center winding alone.



Black-Clawson Co. Airevac

Drive—The d.c. sectional electric drive powers the various components of the coating line.

Console control panels are provided for the coater, drier, and winder assembly. The operator controls the speed of each unit, the overall speed, tension in the web, and the heat in each section of the drier from these panels. Speed adjustment is stepless throughout the entire operating speed range. Dynamic braking is available for emergency stops in addition to normal smooth controlled tension stops. Other controls are provided for run, thread, and individual crawl (for wash-up).

The new facility has an extensive, up-to-date coating preparation system including all the modern testing equipment necessary for checking the variables involved.

Testing equipment located in a special test room includes a forced draft drying oven, muffle furnace, Brookfield LVF viscometer, wall type basis weight scale, binocular microscope and black light.

With its additional facilities Dilts expects to be able to come up with the answers to many of the problems which exist in the industry. Details of the laboratory and pilot plant contract for customer service work, rates, etc., can be obtained by contacting the Black-Clawson Co., Dilts Division Sales Department at Fulton, N. Y.

Company to Manufacture Airevac

Black-Clawson has signed a license agreement with S. D. Warren for the exclusive manufacturing and sale of the patented Airevac unit, which removes air from coating solutions.

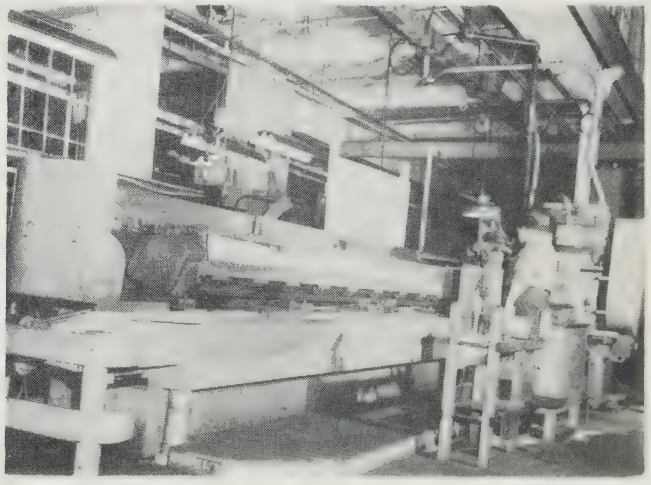
Several Airevac units are available on a rental basis. Write to Black-Clawson Co., Dilts Division, Fulton, N. Y., for full details.

JOHN W. BOLTON & SONS, INC.

New Grinder Handles Extremely Thick or Thin Knives Up to 180 In. Long

To increase its capacity for handling long knives, the company has installed a new, heavy-duty Hanchett grinder capable of handling extremely thick or thin knives of a wide variety of alloys up to 180 in. in length.

The grinder chuck, which was custom-made by O. S. Walker Co. to Bolton specifications, is the first magnetic chuck ever built that is mounted on trunnions or pins with precision bearings. This assures accurate control within a tolerance of 0.001 in. for grinding in any position or angle. It represents an advance in the chuck field, also, because it is electro-hydraulically clamped in position, eliminating the hazard of chuck vibration.



New, heavy-duty Hanchett grinder will accommodate machine knives of any thickness up to 180 in. long at Lawrence, Mass. plant of John W. Bolton & Sons, Inc.

The chuck can be regulated to operate at full strength or any degree of magnetism desired. This means that the grinder can be loaded to its entire capacity while magnetism is controlled so that an individual knife can be removed without disturbing the entire load. Magnetism can be reduced gradually to the point where just enough is applied to hold the piece, which can then be removed manually.

The grinder has a 75-hp. capacity for rough grinding and is also capable of extremely close tolerance work, imparting a fine finish of better than 16 microinches. A special grade of grinding wheel, 32 in. diam. was developed to utilize fully this high power potential.

Bolton Award Winners

Robert E. Lee, Jr. (for the second year in a row), quality control supervisor, Rochester Paper Co., Rochester, Mich.; **Harry "Hop" Bowser,** manager of sales services, Dairypak Butler, Inc., Cleveland, Ohio; **H. S. (Jim) James, Jr.,** director of public activities for the Camp Division of Union Bag-Camp Corp., Franklin, Va.; and **Thomas H. Schumann,** industrial relations director for the Leominster Division of the Mead Corp., were named winners in the 14th Annual Bolton Award Essay contest. They each received a check for $200 and a hand-engrossed laminated wall plaque at different presentation ceremonies.

The subject of this year's competition, which is cosponsored by the Paper Industry Management Association and John W. Bolton & Sons, Inc., was "Privileges of the Free Enterprise System."

THE BORDEN CHEMICAL CO.

Adhesives and Chemicals Publishes Report

A new technical report describing advantages of bulk handling systems, including both receipt and mechanical transfer of packaging glues and resins, is available from the Adhesives and Chemicals Division of Borden.

The report, Number GL-5, discusses cost savings, consumption surveys, design considerations, gives a check list of physical properties, and a complete schematic drawing of an in-plant bulk handling system.

For a free copy of the report write to Department H, The Borden Chemical Co., 350 Madison Ave., New York 17, N. Y.

Aqueous Foil Laminating Adhesive

Availability of a special report on aqueous foil laminating adhesives, based on 15 years of foil adhesive development, has been announced by the company.

The eight-page, 6000-word report incorporates a complete discussion on the physics of foil bonding, including mechanical attachment and polar adhesion; required adhesive properties for foil applications, and analysis of currently used foil adhesives, including neoprene-casein, butadiene-styrene, and nitrile latices, vinyl resins, and acrylic polymers. It discusses foil adhesive problems and lists recommended solutions, including topics such as foaming in applicator tray, foil corrosion due to adhesive film moisture, inadequate adhesive setting speeds, and discontinuous adhesive film at interface. It also deals with techniques of retarding paper curl, and end-use of the foil laminate as a factor in adhesive selection. Technical data for all foil adhesives is reviewed with a listing of solids, viscosity, pH, weight per gallon, and other properties, plus a table showing the effects of dilution upon viscosity.

For a free copy write Department H, The Borden Chemical Co., 350 Madison Ave., New York 17, N. Y.

Appointment

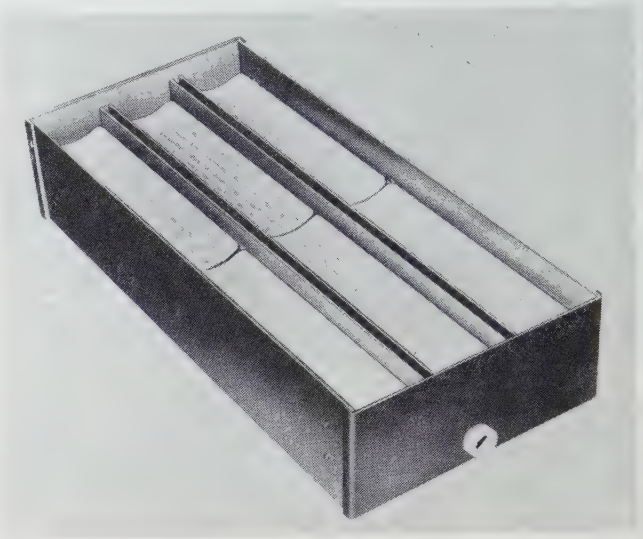
William E. Driscoll has been appointed Midwest District sales manager of the Polyco Monomer Department. He joined Borden Chemical as a salesman in 1956. He is a member of TAPPI and the Paint, Varnish, and Lacquer Association.

COMAC ENGINEERING, INC.

Selected Infrared in High-Speed Drying

The company announces an improved infrared drying oven capable of increasing production speeds in a minimum of space for webs from 10 to 300 in. wide.

Research by Comac Engineering confirmed the fact that materials to be dried, heated, cured, or bonded on the web, respond with different results according to the infrared wavelength employed. For example, in the case of paper coatings, when the correct wavelength is used it will produce maximum evaporation without pinholes, skin hardening, or overheating the paper.



Comac Infra-Air oven, combining selected infrared wavelengths with jet streams of heated air for greater speed in drying, heating, curing, bonding

Comac Infra-Air ovens combine selected infrared energy with hot air jet-streams. This patented feature accelerates evaporation by breaking through and carrying away the vapor layer which tends to cling to and follow the web.

Application information on how to apply infrared energy successfully to paper and paper converting processes for higher production speeds are available without obligation from Comac Engineering, Inc., 239 Mill St., Byram, Conn.

COMBUSTION ENGINEERING, INC.

New Modular Design Boiler for Capacities up to 250,000 lb. per hr.

Combustion Engineering has introduced a new design in its Type VU Boiler series designated as the UV-60. Completely self-cased, the VU-60 is an all-welded, gas-tight envelope of finned furnace and boiler tubes suitable for pressurized or induced draft firing.

Modular design makes it possible to assemble custom designs for standard components.

The VU-60 is available in capacities from 100,000 to 250,000 lb. steam per hr. at design pressures of 250, 500, 750, and 1000 p.s.i.g. Steam temperatures to 900°F. are standard with a built-in flat curve of steam temperature versus boiler rating. Firing may be either horizontal (front wall) or tangential (corners) of oil, gas, or both oil and gas.

Copies of a new brochure "The New C-E Modular VU-60 Boiler" may be obtained from Combustion Engineering, Inc., 200 Madison Avenue, New York 16, N. Y.

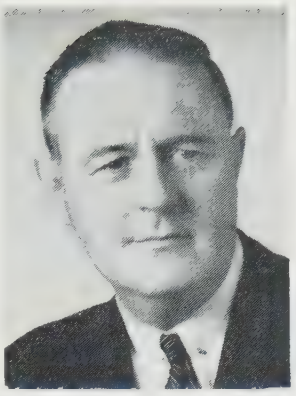
CORN PRODUCTS SALES CO.

New District Manager

Clyde D. Woodburn has been named manager, Philadelphia District Office, Industrial Division, Corn Products Sales Co.

Henry Heinstadt, present Philadelphia manager, is retiring after 43 years of service. Mr. Woodburn, formerly sales-service manager for the company's Eastern region, will assume his new duties immediately.

Mr. Woodburn will have overall responsibility for sale of the company's extensive line of industrial corn starches, dextrins, syrups, and dextrose sugar in southern New Jersey and eastern Pennsylvania.



Clyde D. Woodburn, Corn Products Sales Co.

Don-Oliver Inc.

Dorcon Formed to Manufacture Tanks and Vessels

Dorcon Inc., a wholly owned subsidiary of Dorr-Oliver Inc., Stamford, Conn., is being formed to engage in the design, manufacture, fabrication, and field erection of metal tanks, water and chemical storage facilities, and process and reaction vessels.

Officers of the new company will be President **Alex Fino,** Treasurer **K. Earl Abel,** and Secretary **John W. Swick.** Administrative and engineering offices will be at Warren, Penn., and sales headquarters in Pittsburgh, Penn., with **Max G. Walker** as sales manager.

Dow Chemical Co.

Dry Strength Resin

A resin to improve dry strength has been developed by Dow for the paper and paperboard industries.

Dow said Mydel 550, a copolymer of acrylamide and acrylic acid, is particularly effective on board chemical pulps and groundwood pulps, showing a significant increase in burst, and improvement in tensile, fold, and pick.

With mechanical pulps, the resin demonstrated effectiveness in improving the dry strength in mixtures above a sulfite-groundwood ratio of 50%.

It helps in fiber and filler retention, and improves rosin retention, offers lower steam cost in drying by improving drainage on the wire, and in paperboard production also produced improvements in the bending and stiffness characteristics of the board along with improved bonding.

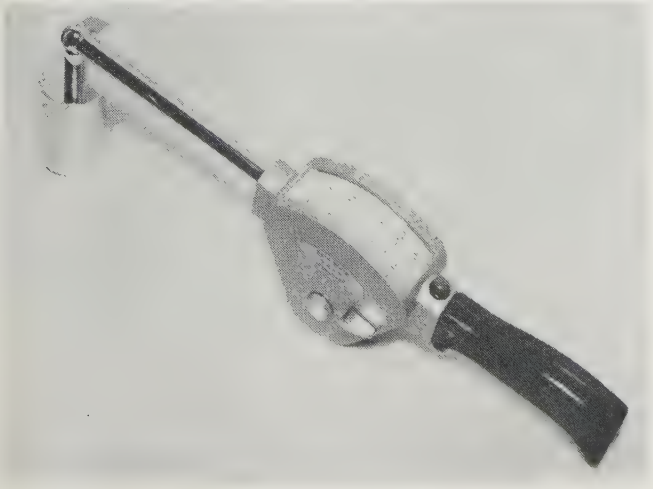
Epic, Inc.

Radiation Pyrometer Kustotherm

The Kustotherm is a surface radiation pyrometer for contactless measuring of temperature of paper and any other nonmetallic materials. It can be used on surfaces of any color, including white, within the limit of permissible error. The swivel radiation head can be directed against vertical, horizontal, or slanted surfaces.

The minimum diameter of the measuring surface is 2½-in. at a measuring distance of approximately ⅓-in.

The range of the instrument is 125 to 480°F. The parallax-free reading on a large scale is almost instantaneous, 4 sec. are required for adjustment. The instrument does not require any batteries or any other power supply, it is easy to operate, and weighs only 2 lb.



The Epic Kustotherm

Fischer & Porter Co.

Bulletin Recommends Bleach Plant Instrumentation

The three-page bulletin recommends the instruments required for a typical three-stage bleaching process—chlorination-alkaline extraction-hypochlorite bleach.

A three-page foldout section, is a detailed flowsheet of the bleach plant, showing the point at which each of the recommended instruments fits into the process.

F & P lists the bulletin as No. 90-26-16. Copies are available from Fischer & Porter Co., 945 Jacksonville Road, Warminster, Pa.

Bulletin of Instrument Symbols for Flow Sheets

A four-page reference bulletin entitled "Suggested Flowsheet Instrumentation Symbols," published by F & P Co., Warminster, Pa., lists the symbols used in drawing flow sheets and schematic control systems for process piping and lines, solenoids and other valves, measuring elements, and miscellaneous process equipment. In most respects, the symbols follow the recommended practice outlined by the Instrument Society of America.

Copies of Bulletin 91-53G-02 are available from the company, 946 Jacksonville Road, Warminster, Pa.

General Electric

Technical Reference on Silicone Fluids

The Silicone Products Dept. of G. E. offers a new, comprehensive technical reference which describes the broad range of major silicone fluids.

Designated S-9, the 20-page, two-color publication differentiates between available grades of silicone fluids, according to viscosity and classifies them on the basis of physical properties. An index permits the user to select a particular fluid on the basis of properties required in his application.

Copies are available on written request to the department, Waterford, N. Y.

New 300-V. Industrial Relays

Bulletin GEA-7329, 12 pages, describes G. E.'s new 10-amp., 300-v. industrial relay designed to cut significantly control panel space requirements. Publication discusses elimination of the need for a wiring trough, one of the major factors in reducing space needs.

For further information write to G. E., Schenectady 5, N. Y.

The Glidden Co.

Appointments

The company's Chemicals Division, Pigment and Color Department, has named **William J. Barnett** sales manager—pigments, with headquarters at the Adrian Joyce Works, Baltimore, Md. **John Owen Jones** was named product manager—colors, with headquarters at the St. Helena Works, Baltimore.

Mr. Barnett, a Glidden sales executive for nearly 20 years, was sales manager of the division's Southern

District at Atlanta, Ga. Mr. Jones had been district sales manager in Cleveland, Ohio.

GOULDS PUMPS INC.

Two Sizes Added

The addition of two new sizes more than double the capacity range of Goulds Model 3305-06 2-stage centrifugal pump line. Now available in sizes 6 × 8–17 and 8 × 10–17 these new units provide capacities up to 3000 g.p.m. for heads up to 550 ft. at 1750 r.p.m. Complete information including performance curves, material specifications, interchangeability charts, and dimensions in Bulletin 722.6 available from Goulds Pumps, Inc., 227 Black Brook Road, Seneca Falls, N. Y.

Chem Process Pumps

Information on bearing life expectancy, shaft deflection, interchangeability charts and diagrams, as well as the usual complete performance, application, and dimension data is included in the revised edition of Bulletin No. 725.1 describing the new line of chemical process pumps recently introduced by Goulds.

Model 3195 is built in eleven sizes covering the range of 5 to 775 g.p.m., 10 to 300 ft. TDH. and are normally stocked in ductile iron, 316 stainless steel, and Gould-A-Loy 20 to give quick shipment on units required for piping a wide variety of corrosive liquids at temperatures from −350 to +500°F. A high degree of interchangeability between pumps and parts, back-pull-out design, and cooling jackets for bearings and stuffing box are outstanding features.

For a copy of Bulletin No. 725.1 write to Goulds Pumps, Inc., 225 Black Brook Road, Seneca Falls, N. Y.

GRAVER WATER CONDITIONING CO.

New Literature on Automatic Filter

A new bulletin on the Graver Monovalve filter, a fully automatic gravity filter with a single, simple control valve, is now available from Graver Water Conditioning Co., New York, a division of Union Tank Car Co.

The literature, Bulletin WC-130, describes the construction, operation, and applications of the Monovalve filter, which stores its own backwash water and is available in single or multi-compartment units. Operating on the loss-of-head principle, which assures automatic backwashing of the filter beds whenever they reach a predetermined "dirtiness," the Monovalve filter requires minimum maintenance and attention and provides a reliable source of uniformly pure filtered effluent at all times.

Bulletin WC-130, which contains flow diagrams and dimensioning data, is available from Graver without obligation. For further information, please contact: Sales Promotion Manager, Graver Water Conditioning Co., 216 West 14th Street, New York 11, N. Y., WAtkins 4-2321.

HERCULES POWDER CO.

Moreland to Distribute Synthetic Resins

Moreland Chemical Co., Spartanburg, S. C., has been appointed a distributor of synthetic resins by the Pine and Paper Chemicals Department of Hercules.

Moreland, who also handles Hercules' pine chemicals, will be distributors of synthetic resins for protective coatings, inks, adhesives, floor tiles, and other applications.

Appointments

Six managerial and supervisory appointments in Plastics Sales, Polymers Department of Hercules, have been announced. The appointments are: **Emmett E. Hixon,** manager, plastics sales; **Robert C. Clark,** product manager of Hi-fax high-density polyethylene; **Robert D. Ullrich,** product manager of Pro-fax polypropylene; **George M. Taylor,** product supervisor of Penton chlorinated polyether; **William O. Bracken,** supervisor, market development; and **Russell D. Hanna,** supervisor, technical service.

With the exception of Mr. Hanna, whose headquarters are at the Parlin, New Jersey Plastics Sales Service Laboratory, the men will continue to be located in the company's Home Office in Wilmington.

The establishment of district managers for plastics sales in five districts has also been announced by the Polymers Department of Hercules. The new appointments, effective immediately, are: **Charles A. Borton,** district manager, Cleveland; **John L. McKeen,** district manager, Chicago; **Paul J. Metzger,** district manager, West Coast; **Daniel G. Welsh,** district manager, Cincinnati; and **Robert R. Stover,** district manager, New York.

District sales of the department's materials for protective coatings will continue to be under the direction of the present manager in each of the five locations.

THE HILTON-DAVIS CHEMICAL CO.

Optical Brightener for Paper

Hiltamine Arctic Paper White, a new optical brightener designed especially for use in paper manufacture has been made available by Hilton-Davis, a division of Sterling Drug Inc.

It is supplied as a clear, odorless, neutral solution containing no flammable solvents. Being predissolved, the new agent eliminates the specking and unevenness of finish often encountered with brightener powders. It is stable to acid and alkali at a pH from 2 to 12, and is compatible with most common paper additives, including starch and rosin sizes.

Samples and technical brochures may be obtained by writing to The Hilton-Davis Chemical Co. Division, 2235 Langdon Farm Road, Cincinnati 37, Ohio.

HOOKER CHEMICAL CORP.

Fact Bulletin on 108 Chemicals

The 1961 edition of "Hooker Chemicals," a 16-page bulletin listing and describing the firm's 108 organic and inorganic chemicals for industry and agriculture, has been published by Hooker.

Copies of Bulletin 100-E are available on request to research, engineering, production, purchasing, and other interested executives, from Hooker Chemical Corp., Niagara Falls, New York.

HODAG CHEMICAL CORP.

Silicone Antifoams Brochure

Many industrial foaming problems are economically solved by the application of small quantities of silicone antifoam agents, reports Hodag, in a new product brochure. The eight-page, illustrated booklet also details the suitability of these Food and Drug Administration sanctioned antifoams in food processing and packaging.

The new brochure may be obtained by writing to Hodag Chemical Corp., 7247 N. Central Park Ave., Skokie, Ill.

E. F. HOUGHTON & CO.

New data sheet describes Cerfak 556, an economical, multipurpose new liquid detergent for deinking, felt washing, system cleaning, and pitch control. More information from E. F. Houghton & Co., 303 W. Lehigh Ave., Philadelphia 33, Pa.

HUYCK CORP.

Alabama Plant Marks Fifth Anniversary

The world's largest modern papermaking felt mill, Huyck's Aliceville, Alabama plant marked the fifth anniversary of its

opening recently. In a week long celebration, employees of the mill together with the townspeople engaged in a variety of activities including a parade, barbecue, band concert, and horse show. In what was referred to as a return to "hard cash," Huyck's payroll for the anniversary week was paid with fifteen thousand silver dollars.

Huyck executives from both Rensselaer and New York City visited the plant to participate in formal ceremonies in recognition of this milestone in the history of the corporation.

Charles LaDow, Aliceville plant manager, expressed appreciation for the contributions made by employees of the plant over the past five years, and he pointed out that much of the credit for the high operating efficiency of the Aliceville Plant belonged to the employees.

Frederic A. Soderberg, general manager of the Huyck Felt Co. Division outlined the company's plans for the future and

L. to r.: Charles LaDow, Aliceville's plant manager, Roderick T. Clarke, secretary of the Huyck Corp., Frederic A. Soderberg, general manager, Huyck Felt Co. Division—and pile of $15,000 in silver dollars

reported that with the expenditure of more than $2,000,000 for new equipment and plant renewal at both plants during 1960–61, Huyck promises even greater operating efficiency in the future. Because of this, according to Soderberg, Huyck's customers will receive greater service. Soderberg also pointed out that Huyck's continuing emphasis on research and development will contribute to the improved efficiency of papermaking felts and to the greater efficiency of the papermaking industry.

INDUSTRIAL NUCLEONICS CORP.

New AccuRay Mark V, Series "E" Equipment

The company announces the introduction of newly designed remote equipment cabinets, and, pedestal, wall mounted, and floor mounted operator's control stations. The new design will be designated as AccuRay Mark V, Series "E" equipment.

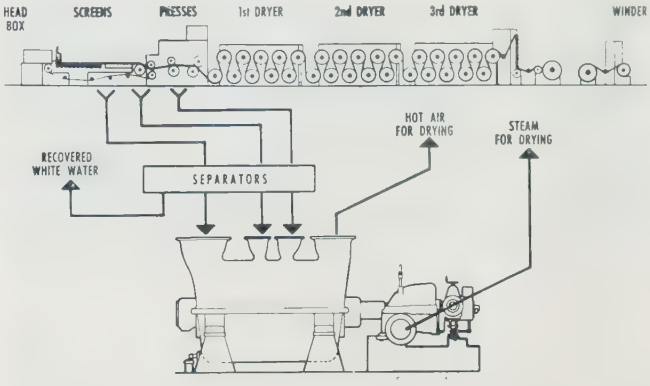
The Mark V, Series "E" equipment cabinets contain all electronic equipment and controls not specifically required in the production area. Within the cabinets, separate functional panels are mounted on rugged cast frames which swing out to allow easy access to every panel and component in the system. Also, a complete system drawing following J. I. C. Standards is mounted on the inside door as a maintenance aid.

The AccuRay Mark V, Series "E" Operator Stations house only those controls required by the machine operator. Since the stations are usually located near processing machinery care has been taken in the design to protect components against undesirable environments.

INGERSOLL-RAND CO.

Central Vacuum System for Paper Mfg. Industry

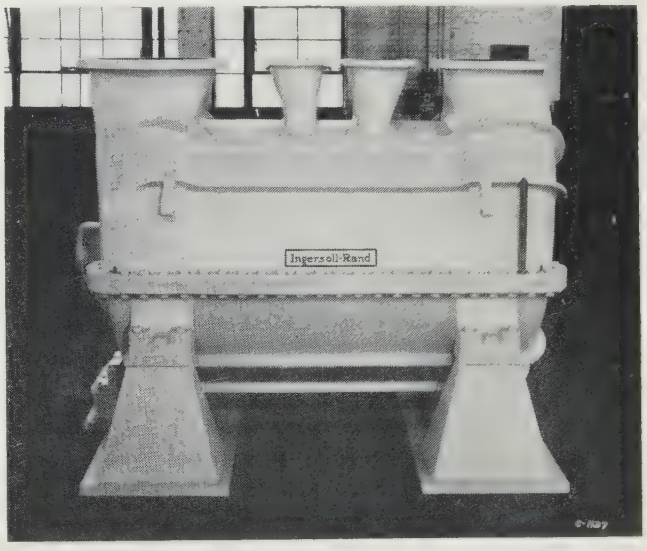
A central vacuum system for papermaking machines, the first of its kind in the United States, has been demonstrated by the company at its Phillipsburg, N. J. plant. The system is designed to handle all the vacuum requirements for an entire paper machine, and will cut annual operating costs by



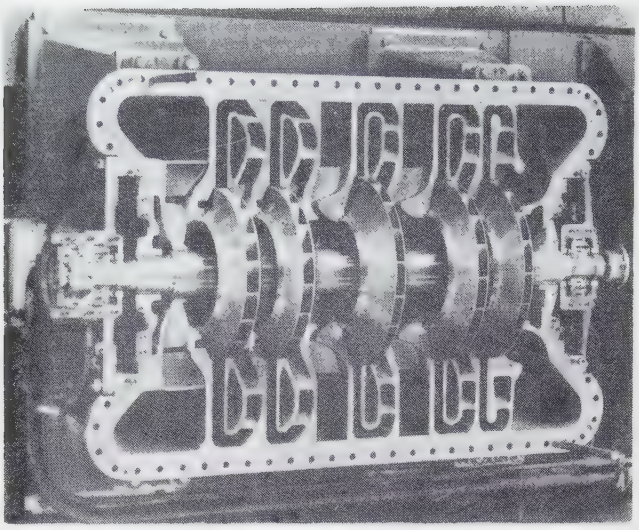
Simplified flow diagram. Because it handles all of the vacuum requirements for an entire paper machine, the Ingersoll-Rand central vacuum system becomes an integral part of the papermaking process. The system, which is engineered to meet the specific requirements of the installation, features a single centrifugal exhauster which provides vacuum at various levels

more than $25,000, according to J. A. Wiendl, vice-president and domestic sales manager for Ingersoll-Rand. A central centrifugal exhauster with multiple inlets provides vacuum at various levels, and the exhauster can be driven by either electric motor or steam turbine.

The demonstrations were conducted in the company's compressor testing department where the system was set up to operate under simulated paper machine conditions. The system operated at three different design points and was put



Centrifugal exhauster. Side view of the centrifugal exhauster showing the arrangement of the three intake connections and the single discharge connection. The 20 Hg vacuum connection is at the left, followed by the 15 in. Hg and the 10 in. Hg connections. The discharge connection is shown at the right. All the connections can be arranged in the up position, as shown, or in the down position, depending on the installation. The casing is horizontally split



Exhauster design. This view of the Ingersoll-Rand exhauster with the top-half casing removed shows the internal construction with the single moving element—the rotor. Bearing boxes are outside of the air-flow passages, assuring that no lubricating oil can get into the discharge air stream. The hot, dry discharge can then be used without danger of contaminating even the finest papers

The general layout of the new Koppers Research Center in Somervell Park, Monroeville, Pa., is shown in this view from the northwest, or main entrance to the Park. At the left is the library and cafeteria wing. The entrance and lobby are just left of the tall building in the center which houses all administrative offices and a spacious auditorium. At the right are typical laboratory wings, projecting alternately north and south from a glassed-in corridor which connects the administration building to the wings. At the near end of the closest wing is one of the glass enclosed steel stairways, typical for each laboratory wing, which may be removed if it should be necessary to extend the length of the building

through a simulated "paper-break" demonstration. Test facilities were highly instrumented so that the performance of system components could be checked by those attending the tests. This system does the work of six or more individual vacuum pumps and reduces vacuum horsepower requirements for a paper machine by as much as 25%. Heat recovery is equivalent to 70 to 90% of the driver horsepower and can be used for heating and drying applications throughout the mill.

JOHNS-MANVILLE

Pressure-Sensitive Tapes

"How to Select a Pressure-Sensitive Tape" is the title of a helpful booklet published by J.-M. Dutch Brand Division. The booklet, DB-56A, contains information on the types of tapes available, how they are constructed, and the factors involved in choosing a particular tape for a specific application.

Copies of DB-56A can be obtained free on request from Johns-Manville, Dutch Brand Division, 22 East 40th Street, New York 16, N. Y.

KOPPERS CO., INC.

Somervell Park Research Center

Koppers dedicated its newest and largest research facility, at Somervell Park, Monroeville, Pa. on August 28. It represents an investment of about 8.5 million dollars. Koppers Co., a 49-year-old company with annual sales in excess of 300 million dollars, began to accelerate its research program about fifteen years ago when it entered the field of chemicals and plastics. Until that time the company had been identified with engineering and construction—mostly in the steel industry; coal by-products, wood preserving, fabricated metal products, and merchant coke plants.

Only about one-third of the building planned for the 176 acre site has been completed. The center will eventually house over 1500 scientists and their assistants—about four times the present population.

Among the many research and development accomplishments a number of interests were demonstrated at the recent open-house celebration. These included Dylite, expandable polystyrene, a foam plastic introduced into this country by Koppers, chemically impregnated Non-Com fire retardant wood products, which have opened up major new uses for wood, particularly in commercial and public buildings where fire safety is important; a new line of polyepoxide resins with potential markets in the coating and adhesive fields and in insulation of electrical components. They are known as KER epoxy resins and will be introduced this fall; development of a superior, high velocity irrigated precipitator which removes waste particles from industrial stack gases; many contributions to the increased efficiency of steelmaking, including the development of better coke and more efficient utilization of low grade iron ores through pelletizing.

Among the areas of particular promise is work in the field of boron chemistry, plastics for use as building materials, fiber reactive dyes, chemical intermediates, coloring of anodized aluminum, and the desalinization of sea water. This is known as the Koppers hydrate process, the only chemical approach to the problem now being made.

The Center itself consists of a 5-story administration building, three 3-story interconnecting laboratory wings, and several outlying auxiliary buildings. A feature of the Center which contributes to its easy expansibility are the glass enclosed stairways at the end of each wing which can be easily removed when additions are called for, and reaffixed to the end of the new wing.

SAMUEL M. LANGSTON CO.

Langston Moves All Printer-Slotter Operations to Camden, Dissolving Greenwood Eng. Co.

Langston announces it is moving manufacturing of all Greenwood printer-slotters and parts from Baltimore, Md., to its Camden, N. J., plant and dissolving Greenwood Engineering Co., a wholly owned subsidiary.

The move, effective September 1, will also transfer to the Langston organization a number of key sales, engineering, and production people from Greenwood.

President Bryant W. Langston said consolidation of all work on printer-slotters and corrugating equipment in one plant would increase efficiency and enable the company to continue to keep prices down.

LUNKENHEIMER CO.

"King-Clip" Gate Valve Circular

Lunkenheimer "King-Clip" gate valves, designed to with-

stand corrosive actions too severe for lighter valves, are described in newly issued Circular 561 from Lunkenheimer.

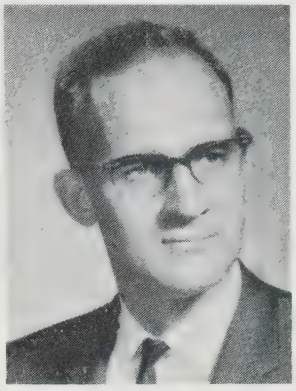
Reports are given on the applications, features, and designs of the "King-clip," originated by Lunkenheimer. This valve is made in two types—the iron-body, bronze-mounted for general corrosive service; and the all-iron for solutions which attack bronze but not iron.

Steam pressure and water-oil-gas pressure ratings are listed, as well as specific dimensions for different "King-clip" designs. All designs are illustrated, including a cutaway for parts identification.

Circular 561 may be obtained by writing to the Lunkenheimer Co., Cincinnati 14, Ohio.

MANCHESTER MACHINE CO.

John R. Hanzlik has been named manager of sales service. This is a new position recently created under Manchester's expanding sales organization program. Mr. Hanzlik is primarily responsible for home office coordination of order processing and for providing support to the field sales engineering organization. In this capacity, he will prepare equipment proposals and specifications, coordinate order scheduling activities and other customer service functions of all kinds.



John R. Hanzlik, Manchester Machine Co.

MIDLAND-ROSS CORP.

Expansion of Overseas Operation

M-R has negotiated an agreement with Ameliorair, Société Anonyme in Paris, France, for the manufacture and sale of a broad range of products of the J. O. Ross Engineering Division of Midland-Ross. The Cleveland-based firm also will acquire an equity interest in the French company whose present major product lines include textile machinery, industrial ovens, and air-conditioning equipment for industry. Ameliorair has four plants in France.

Wade N. Harris president of Midland-Ross, noted that the complete product lines of the company's Waldron-Hartig and Ross Divisions now have been licensed for manufacture in Europe. Earlier this year, Midland-Ross made an agreement with Henry Simon Limited in England for the manufacture and sale throughout Europe of the Waldron machinery products of its Waldron-Hartig Division.

Distributors and sales representatives at MCP's 8th Technical Conference

MINERALS & CHEMICALS PHILIPP CORP.

MCP Distributor Training Conference Emphasizes Research

Over 50 distributors and sales representatives attended a four-day technical conference recently held by MCP at its Menlo Park, N. J., offices and research laboratories. An annual affair, the meeting this year was planned as a "refresher course" with specific emphasis on MCP's close sales ties with its research department.

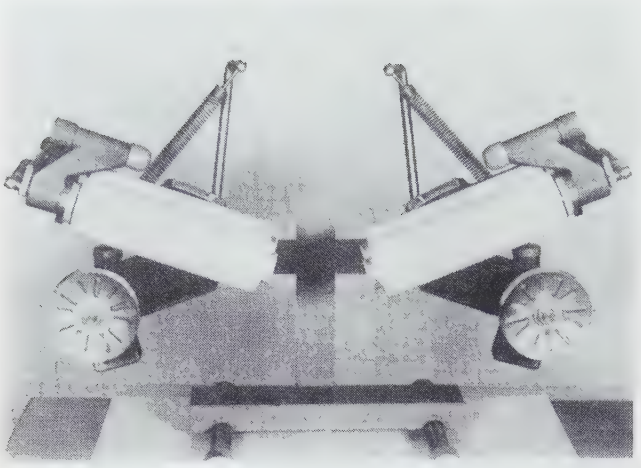
To establish a thorough understanding of MCP products, each session included a description of the raw basic material as mined, how it was processed, its properties and possible applications after processing, and possible future applications. Descriptions of specific uses in various industries were offered by representatives of MCP customers. A special trip to one of the most modern corrugating plants in the United States was made to show at first hand how one of MCP's latest products, Corragel, was performing in corrugating starch adhesives.

MOUNT HOPE MACHINERY CO.

Two Lower-Cost Models of Precision Guiders for Textiles, Paper, and Film Added to Mount Hope Line

Two new models of the Mount Hope Precision Guider will mean cost-savings for manufacturers who do not have severe acid conditions in the processing of textiles, paper, and plastics, according to officials of Mount Hope Machinery Company, Taunton, Mass. The new units—Models "A" and "B"—perform in exactly the same manner as the standard Model "S" stainless steel guider, assuring accurate lateral alignment of materials ranging from the heaviest of fabrics to the lightest, most sensitive of films. They are less expensive because they are fabricated of aluminum and cadmium-plated steel.

New Precision Guiders added to Mount Hope line



Mount Hope guider

Now, Mount Hope engineers say, the standard stainless steel Precision Guider is available for severe wet acid service; Model "A," with aluminum frame and stainless steel accessory parts and nip roll denippers, is geared to wet and mildly acid service; and model "B," which is fabricated of aluminum but has some accessory parts made of cadmium-plated mild steel, is designed for dry or damp service. Economy Model "B" does not have denippers.

Precision guiders were the first web control devices designed and manufactured by Mount Hope Machinery Company and are the products on which the company originally was founded. Continued rise in steel costs, coupled with technological advancements in the uses of aviation aluminum, brought about the development of the newer models. Mount Hope engineers feel the less-expensive units will prove useful to textile, paper, plastics, and rubber companies.

Mount Hope Precision Guiders provide instantaneous detection of misalignment in processing and automatically adjust for the indicated deviation by tilting a pair of nip rolls—one pair for each selvage. The nip rolls exert a gentle pushing and pulling action on the selvage accomplished by the gradual tilting of the rolls to hold the web in alignment. The sensing element is a grooved and fluted selvage wheel which controls the angle of the nip roll. The web travels between these rotating wheels which hold and control the selvages gently by means of spring pressure.

There are two principal advantages to the Mount Hope guiders:

1. Because the selvage is always in contact with the roll, the rolls are always rotating. This eliminates the possibility of fraying, which can occur when contact is lost. When this happens, the rolls stop turning, and must be started again with attendant friction when contact is made.

2. The guiders rotate through a continuous rather than a fixed angle. Therefore, the angle of compensation is exactly what is required—no more, no less. This prevents "jerky" corrective action which can occur when a fixed guider is set for maximum deviation.

The strictly mechanical devices assure minimal maintenance—there are no electric switches or coils to burn out and no air valves to clog or leak. All moving parts are ball-bearing mounted. Bearings are waterproofed and are fully protected by deflecting shields to allow ease of machine washdown.

The guiders operate from any angle, in any position, on any flexible material of any width. They are available with a variety of guider stands—floor, bracket, laterally adjustable bracket, tenter, and rack.

National Starch and Chemical Corp.

New Monomer Plant

National Starch has announced plans to build a $4 million vinyl acetate monomer plant with an annual capacity of 45 million pounds.

One of two sites currently under study in Texas will be selected for the location of the plant. Construction, which will be handled by Brown and Root, Inc., is scheduled to commence late this year, and the new facility is expected to be in operation during the fall of 1962.

Nopco Chemical Co.

Bulletin on Processing Chemicals Described in Nopco Chemical's Bulletin

A condensed listing of the more important processing chemicals available from its Industrial Division has been published by Nopco.

Categories of products included are surfactants, hydrosulfites and sulfoxylates, defoamers, resin and wax emulsions, water-soluble lubricants, water-soluble resins, softeners, and gas-fading inhibitors. Also listed are enzymes, metallic soaps, stabilizers, and inorganic compounds.

The new bulletin, No. ISP-50, may be obtained by inquiry to Literature Department, Nopco Chemical Co., 60 Park Place, Newark 1, N. J.

Oakite Products, Inc.

Appointment

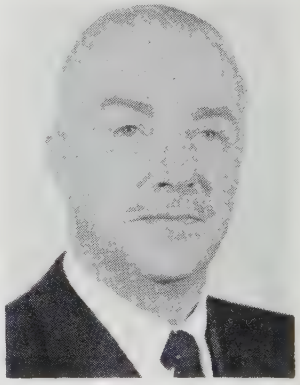
Robert P. Jones, New England division manager of Oakite since 1954, has been assigned to the New York headquarters of the firm, which manufactures industrial cleaning and metal treating compounds. Mr. Jones, whose experience includes purchasing and accounting as well as sales, will do staff work for the firm, reporting directly to the president, John A. Carter.

Penick and Ford Ltd.

Pacific Sales Territory

The creation of an autonomous Pacific sales territory by the Corn Refining Division of P & F was announced by O. H. Tousey, corporate vice-president. The Pacific area had formerly been a district of the Western territory.

Harold A. Harvey, district sales manager for the Pacific Coast, was appointed sales manager of the territory.

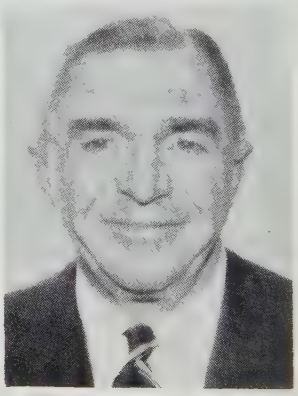


Harold A. Harvey, Penick & Ford, Ltd.

Appointments

James W. Ferguson has been appointed a general trade

James W. Ferguson, Penick & Ford, Ltd.



S. F. M. Maclaren, Penick & Ford, Ltd.

L. S. Simser, Penick & Ford, Ltd.

G. M. Anderson, Penick & Ford, Ltd.

salesman, Corn Refining Division. He will call on food and paper manufacturers in Iowa, Nebraska, Kansas, and Missouri.

S. F. M. Maclaren has been appointed eastern sales manager, replacing **W. S. Russell** who has been promoted to assistant to the vice-president.

L. S. Simser has been appointed western sales manager, replacing **L. S. Poer** who has been promoted to vice-president and director of marketing.

G. M. Anderson has been appointed southern sales manager, replacing **P. G. Wear** who will retire January 1, 1962.

R. I. Bashford, J. R. Heard, and **J. A. Kooreman** are appointed industry managers with broad planning and coordinating responsibilities for activities in specific industries, paper, textiles, and food and allied trades, respectively. This new function is designed to improve general operating efficiency.

Pennsalt Chemicals Corp.

Appointment

Dean E. McCrory has been appointed assistant district sales manager at Tacoma, Wash. He replaces **Ralph C. Schaeffer** who was earlier named district sales manager at Salt Lake City.

Pfaudler Permutit

Literature on Glasteel ClO_2 Generating Plants

A new 6-page bulletin describing the advantages of Glasteel as a material of construction for ClO_2 generating plants is now available from Pfaudler, Co., a division of Pfaudler Permutit Inc.

The opening section of the bulletin describes the physical chemical, and economic chracteristics which particularly suit Glasteel for use in chlorine dioxide generation. Particular emphasis is placed upon the ability of Glasteel to withstand all of the corrosives involved in chlorine dioxide generation including ClO_2 itself.

A two-page, fold-out, flow chart shows all of the basic components involved in a typical Pfaudler engineered ClO_2 generating plant. Actual installation photos of Glasteel equipment are inset into the flow chart to provide a realistic picture of size, configuration, and integrated design that has gone into existing installations.

The final section of the bulletin briefly describes the services available from Pfaudler's Project Engineering group in regard to ClO_2 generating plant design, specification, layout, procurement, erection, and startup.

Copies of the new literature may be obtained by requesting Bulletin 1011 from The Pfaudler Co., 1133 West Avenue, Rochester, N. Y.

Pittsburgh Plate Glass Co.

PPG Chemical Division Patents

Patents of interest to the paper industry granted to the company, Chemical Division (formerly Columbia-Southern Chemical Corp., a subsidiary) include two concerning the manufacture of silicate and silica pigments and their use as fillers for paper.

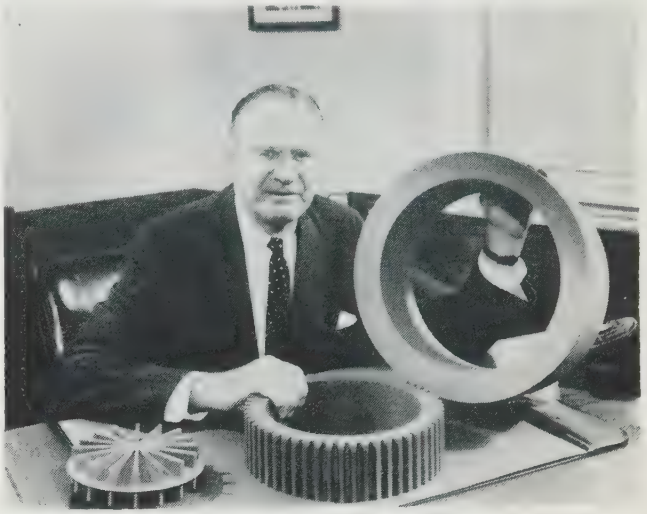
U. S. patent no. 2,943,970 (July 5, 1960) discloses that the effectiveness of a silicate or silica pigment is influenced by the development of a flocculated condition of the pigment, and that the flock size can be controlled to produce the desired results.

U. S. patent no. 2,943,971 (July 5, 1960) discloses the synergistic effect of the joint use of a silicate or silica pigment and titanium dioxide in obtaining optical properties of increased brightness and opacity in the paper above values which could be obtained by use of either pigment alone.

The Polymer Corporation

Massive Nylon Shapes for Papermaking Equipment

An economical nylon casting process makes available to the paper converting industry large nylon shapes for use as roll



MC nylon parts made by a new monomer casting process are examined by Louis L. Stott, president, Polymer Corp.

covers, guides, bearings, gears, and similar parts which require lightweight and maximum wear resistance.

Louis L. Stott, Polymer president, described the new process as "monomer casting," a direct conversion of nylon from basic chemical raw materials to finished parts or shapes. The process is said to be similar in techniques and costs to the conventional casting of metals, and it is expected to open up new markets for nylon in heavy industry now using cast metal parts.

Prior Chemical Corp.

Appointment

Lester C. Lewis has been elected a vice-president of the corporation, the 39-year-old chemical concern. In addition, Mr. Lewis was elected to serve in the same capacity with their affiliate, Payson Corp., both having executive headquarters at 420 Lexington Ave., New York, N. Y.

Rice Barton Corp.

Two Industries Purchased

Worcester Stamped Metal Co. and Armour-Porter Co., Inc. have now become wholly-owned Rice Barton subsidiaries. Both will continue operating at their present sites.

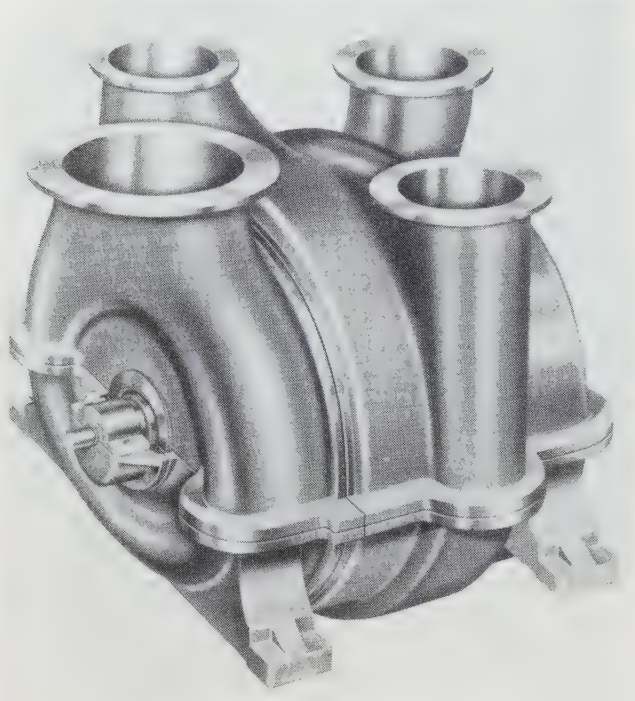
Specialists in steel weldments Armour-Porter makes welded machine bases, guards, jigs, fixtures, and pressure vessels. Incorporated in 1916, Worcester Stamped Metal normally employs 100 people for forming steel, aluminum, and many new exotic metals and alloys in making parts for aircraft, automative, and other industries.

ROOTS-CONNERSVILLE BLOWER, DIVISION OF DRESSER INDUSTRIES, INC.

Multistage Centrifugal Exhausters for Paper Machine Vacuum Service

The company is offering a new multistage centrifugal exhauster which will handle all the vacuum requirements of a paper machine. The unit will provide 20-in. Hg vacuum with interstage connections for the 15 and 10-in. vacuum requirements at the paper machine, plus line, valve, and separator losses between the paper machine and exhausters.

The new multistage exhauster offers several advantages. It incorporates the reliability and low noise level proved in



Roots-Connersville multistage centrifugal exhauster for paper machine vacuum service

Roots centrifugal compressors for other application; centrifugals implicity assures minimum maintenance; a single exhauster and driver will meet all vacuum requirements of the paper machine; floor space and foundation requirements are reduced and piping is simplified; and the compression-heated exhaust air can be used for drying.

Water spray cooling and primary and secondary moisture separators in each suction line assure cool dry air to the exhauster for reduced horsepower requirements. Each vacuum line also includes an automatic control valve to regulate vacuum levels.

The standard exhauster is a four-stage machine. A fifth stage can be included for vacuum levels below 20 in. Hg at the exhauster inlet. Six models of the multistage exhauster are available with capacity ratings from 10,000 to 55,000 cu. ft. per min. air at inlet conditions.

Additional details can be obtained from Roots-Connersville Blower, Division of Dresser Industries, Inc., 900 West Mount Street, Connersville, Ind.

THE RUST ENGINEERING CO.

Senior Paper Consultant Named

William M. Crawmer was made senior pulp and paper mill staff consultant of Rust Engineering. He joined Rust in 1959 as a staff consultant to specialize in the design and construction of pulp and paper mills. Previously, he was a pulp mill superintendent for Mead Corp. with ten years' service.

SANDOZ, INC.

Hans Seifert has been appointed sales manager, Chemical Division. He will be responsible for all chemical sales of the company.

THE SANDY HILL IRON AND BRASS WORKS

Appointment

Oliver A. Laakso, president of Kamyr, Inc., was elected to membership on the Board to fill the vacancy caused by the death of Charles F. Bookwalter.

Sandy Hill has been closely allied with Kamyr for more than 25 years in the sale and manufacture of equipment for the processing of wood pulps.

SHAWINIGAN RESINS CORP.

Gelva PVAc Manual

A comprehensive technical manual on the properties and uses of Gelva, polyvinyl acetate, has been published by Shawinigan.

The 40-page manual contains detailed specifications and physical characteristics on all homopolymer and copolymer resins, emulsions, solutions, and spray-dried powders.

Data are also given on Gelva applications in building products, paints, paper and textile coatings, adhesives, and others.

Copies of the manual may be obtained from Shawinigan Resins Corp., Department RH, Springfield, Mass.

Appointments

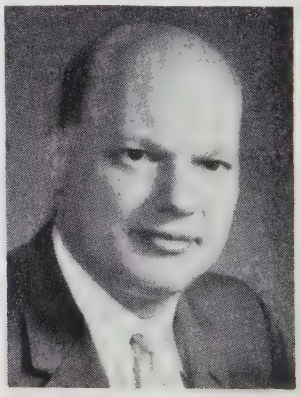
Irving Ash and **Bruce E. Porter** have been appointed to the newly created post of district manager for Shawinigan.

They will be responsible for the activities of the sales staff in considerably enlarged territories. Mr. Ash is located in New Jersey and Mr. Porter in Cleveland, Ohio.

David S. Cox, a member of the company's product development staff, has been named assistant manager of the department.

Appointed technical service managers in their specialized areas have been **David R. Cahill,** paper and textiles; **Stedman C. Herman,** adhesives; and **Robert W. Ross,** electrical insulation. In addition, **John R. Danner** has been transferred to the technical service department as a technical service representative for building products and surface coatings.

Hans Seifert, Sandoz, Inc.



Oliver A. Laakso, Sandy Hill Iron & Brass Works

Herbert Terry, who has been a research section leader, has been appointed technical service manager for building products and surface coatings.

SPROUT-WALDRON & CO., INC.

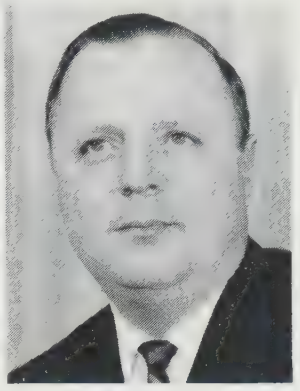
20-Inch Pressurized Refiner

The addition of the newly designed refiner for up to 250 hp. for the smaller tonnage applications is announced by the company.

Their complete line now includes 20-26-34 and 42-in. units. Bulletin 212-B on request from the S-P, Muncy, Penn.

C. W. Kyle to Represent Company

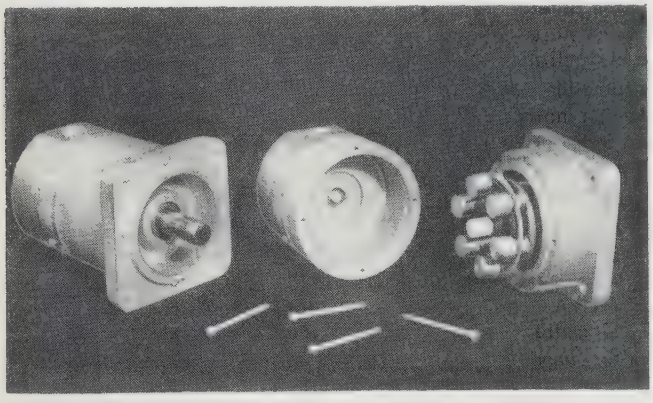
C. W. Kyle has joined the company as a sales representative and will represent it in the sale of its pulp and paper equipment, in Ohio, Indiana, Michigan, and Kentucky. Mr. Kyle will work out of Franklin, Ohio.



C. W. Kyle, Sprout-Waldron & Co., Inc.

SQUARE D CO.

A new Synchro-Snap centrifugal switch with negligible friction to provide repetitive accuracy has been introduced by the EC&M Division of Square D. Developed to replace



Square D Synchro-Snap Centrifugal switch

existing centrifugal switches—which depend on complicated frictional relationships between weights, bearings, bearing shafts, and coil springs—the new Square D switch can be used for speed sensing, overspeed and underspeed detection, speed interlocking, sequencing, and signaling.

For details, write to Square D Co., EC&M Division, 4500 Lee Road, Cleveland 28, Ohio.

U. S. INDUSTRIAL CHEMICALS DIVISION, NATIONAL DISTILLERS AND CHEMICAL CORP.

Responsibility for the operation of the two government-owned chlorine-caustic soda units at Huntsville, Ala., will be assumed by Stauffer Chemical Co. on December 15, 1961. The present leaseholder is U. S. I.

According to R. W. Gunder, vice-president–sales for the Industrial Chemicals Division of Stauffer, the output of the plants will continue to serve the pulp and paper, chemical, textile, petroleum, and bleach manufacturing industries and the public health needs of the Southeast. The plants will continue to be operated with their present personnel.

TESTING MACHINES INC.

Reference Manual for Quality Control and Research

Technical and purchasing executives may now obtain a copy of the new 300 page "TMI Catalog and Register of Testing Machines."

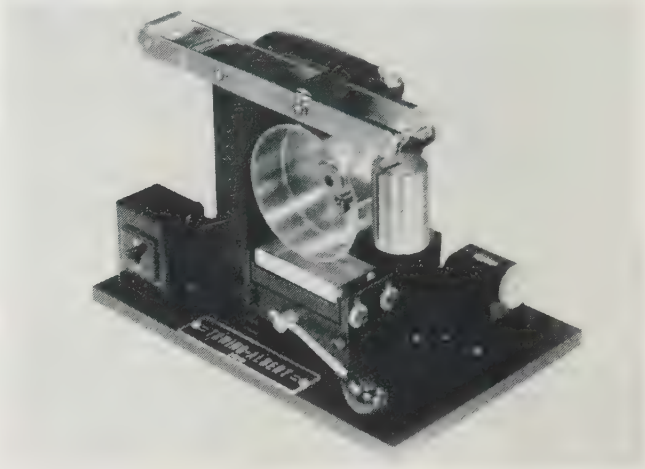
This reference manual costs $6.00 per copy, and includes 1239 physical testing machines for all industries.

The equipment has been compiled from world-wide sources, including units manufactured by Testing Machines Inc., and the products of many other prominent manufacturers here and abroad. The book is a helpful reference source for technical and purchasing executives, educational institutions, and governmental agencies. Distribution will be on a selected basis. Please write to Testing Machines Inc., 72 Jericho Turnpike, Mineola, N. Y., on your company letterhead, indicating your title or position.

THWING-ALBERT INSTRUMENT CO.

New Wet-Rub Tester for Evaluation of Coated Paper

The test method involves wrapping the test specimen around a stainless steel drum and rotating it against an idler



The wet-rub tester

roll covered with smooth-surfaced gum rubber. This provides a slip-free operation.

The effective weight of the idler against the sample may be varied by means of auxiliary weights supplied with the instrument.

The weight of solids removed from the sample during a predetermined number of revolutions is readily obtained by evaporation of the reagent grade water through which the sample rotates during test. Good repeatability is claimed.

The wet-rub tester was developed at the Research Laboratories of Corn Products Refining Company, Argo, Ill. For more information write to the Thwing-Albert Instrument Co., Penn Street & Pulaski Ave., Philadephia 44, Penn.

Facts on Testing

"Facts on Testing" is written and published periodically to inform technical people concerned with physical testing of new developments in test instruments, new testing methods, new applications for test instruments, short cuts discovered by other readers, new useful accessories for instruments, and trends in testing research and development work.

"Facts on Testing" is available from Thwing-Albert Instrument Co. at Penn Street & Pulaski Avenue, Philadelphia 44, Penn.

WESTINGHOUSE ELECTRIC CORP.

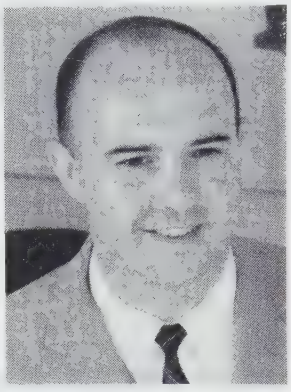
Insulation Increases Oxidation Resistance of Celluloses

Licensing arrangements for using Insuldur® insulation for application to cellulose products to increase their resistance to oxidation, heat, and aging are available from Westing-

house. This new low-cost material will also improve the tensile and bursting strength of paper, cotton fabrics, hard fibers, and other cellulosic materials.

Insuldur insulation is a cellulosic product thermally stabilized by increased oxidation resistance due to the introduction of chemical additives to the material. High-temperature testing has indicated that the stabilized cellulose can operate at a temperature approximately 30°C. higher than regular cellulose and still maintain the same life expectancy.

For further information, write Westinghouse Electric Corporation, P. O. Box 2099, Pittsburgh 30, Penn.



Maurice S. Letourneau, Wica Chemicals, Inc.

Wica Chemicals, Inc.

Maurice S. Letourneau has been appointed general manager, Chemicals Department, Charlotte, N. C.

He will supervise sales of coatings and sizes for paper and of intermediate components for adhesive and paint manufacture, and will be responsible for markets in the eastern and southeastern United States.

General

Engineering Index

The Engineering Index annotates and indexes selectively on the basis of engineering significance, the available current technical periodicals received by, and permanently housed in, the Engineering Societies Library. Included are the regular professional journals, trade journals, publications of engineering societies, scientific and technical associations, universities, laboratories and research institutions, government departments and agencies, and industrial organization. Papers of conferences and symposia, separate and nonserial publications of various kinds, and selected books are also covered. Patents are excluded. In all, over 3300 publications are received. These are printed in 22 languages and come from 42 countries.

The Engineering Index service is divided into 249 "Field of Interest" Divisions. It is issued in two forms: on 3×5 library cards mailed weekly to all subscribers, and in a bound volume, 7×10 inches, which contains the information distributed by the card index service for a full calendar year. The 1960 Bound Volume, 1759 pages, was ready for distribution July 1, 1961, the price is $75 post paid.

Order from The Engineering Index, Inc., 345 East 47th Street, New York 17, N. Y.

Recommended Procedures for Entering Tanks

An eight-page pamphlet on recommended safe practices and procedures for entering tanks and other enclosed spaces is now available from the Manufacturing Chemists' Association, Inc.

The publication emphasizes that hazards inherent in tank entry can be avoided or overcome by following three basic principles. They are:

Establish a definite system of pre-planning for tank entry and a worker instruction program; Prepare the vessel for entry by physically isolating it, cleaning it to remove contaminants, and testing it to insure absence of such contaminants; Use a formal permit system requiring written authorization for entry to be issued only after the supervisor in charge is satisfied personally with tank preparation, precautions to be taken, personal protective equipment to be used, and procedures to be followed.

The pamphlet suggests a sample work permit for employees entering the tank or enclosure and also recommends tools and protective clothing needed by employees.

SG-10 is available from the Manufacturing Chemists' Association, 1825 Connecticut Ave., N. W., Washington 9, D. C. for 30 cents a copy.

New Literature Searching Service for Science and Industry

In response to public requests for "tailor made" bibliographies of technical information suited to the day-to-day as well as long-range requirements of scientists, engineers, and technical administrators, the Office of Technical Services, in cooperation with the Science and Technology Division of the Library of Congress, has established a fast, inexpensive program of searching two major scientific and technical literature sources. A fee of $8 per hour will be charged for searches.

According to the desires of the requestor, searches will be made of *either* or *both* the Office of Technical Services collection and the Library of Congress collection to the extent specified. Two types of searches are offered:

Current Awareness Bibliography Service—The purpose of this service is to keep the subscriber abreast of new literature in his field on a periodic basis. The subscriber outlines the generic and specific subject fields in which he is interested, and at intervals prescribed by him (1 month, 3 months, etc.) he receives references to pertinent reports, periodical articles, technical papers, and other materials accumulated during the period.

Retrospective Bibliography Service—The requestor is furnished a bibliography of material available at the time of the request and in accordance with literature dates and other limitations assigned by him.

Board Production

Estimates made by the American Paper and Pulp Association indicate that paper and board production for the first half of 1961 was 17.4 million tons, 1% below production for the first half of last year. Paper production was 7.8 million tons, a decline of 1%; paperboard was 8 million tons, equal to production last year, and construction paper and board was 1.5 million tons, 5% under last year's first 6 months.

Pelleting of Bark

Successful installation of a unique shredding, drying, and pelleting process which makes it economical and profitable to burn bark for fuel has been announced by Sprout, Waldron & Co., Inc. The system is now in operation at Southern Extract Co., Knoxville, Tenn. It is designed to shred the bark, pellet it, and store it for automatic and continuous metering to the coal bunkers. Estimated savings from the new process are in the area of $17\frac{1}{2}$ tons of coal per day.

The problem of bark disposal is becoming increasingly important to the pulp and paper industry. Hauling of bark to dumps is rather costly and extremely wasteful. Numerous attempts at the production of salable by-products have met with only limited success. In the "as yield state," bark does not burn well, and the heat values hardly justify the expense involved. The bark pellets formed by the new process give burning rates that are reported to be equivalent to the soft coal currently in use at Southern Extract. The Sprout-Waldron system consists essentially of a hammer mill for shredding the bark, a special flash drying system, pelleting equipment and the necessary auxiliary materials handling units for conveying and storing the pellets, as well as for blending the pellets with coal. The operation is continuous and completely automatic, requiring no more than one operator for supervision and maintenance purposes.

DIVISIONS AND COMMITTEES

Authorized Committee Assignments

The following new committee assignments were made during August, 1961. The total number of assignments now active in the 64 TAPPI technical committees is about 400. An identifying number is given by TAPPI Headquarters to each work assignment made by a committee chairman and approved by the appropriate division chairman. Assignment numbers should be requested for *all* activities of technical committees. A separate series of numbers is reserved for each of the technical divisions.

Assignment no.	*Committee*	*Title and objective*	*Committeeman in charge*
		ENGINEERING DIVISION	
4045	Drying	"A Bibliography of Paper and Coating Drying—1960," to prepare an annotated bibliography and summary of the publications in the field of paper, pulp, and coating drying for the year 1960.	S. G. Holt
4046	Industrial Engineering and Materials Handling	"Waste Paper Storage Facilities," to obtain and present useful data on storage space requirements for baled waste paper.	C. H. Reagan
4047	Industrial Engineering and Materials Handling	"Pulp Volumes versus Densities," to present to TAPPI members in useful tabular form the relationship of pulp density to volume.	B. C. Kendall
4048	Industrial Engineering and Materials Handling	"Unit Weight per Cord of Chips," to compile and present reliable data on weight per cord of chips of various species.	H. A. Stoess
4049	Industrial Engineering and Materials Handling	"Lineal Feet of Paper per Roll," to present in chart form a convenient means of obtaining lineal feet of paper in a roll, having roll diameter, core diameter, and basis weight.	C. H. Reagan
4050	Industrial Engineering and Materials Handling	"Roll Weights per Inch of Trim/Diameter," to present in chart form a means of obtaining quickly weight of a roll of paper, having trim width and diameter in inches.	C. H. Reagan
4051	Industrial Engineering and Materials Handling	"Winder Sets per Reel of Various Diameters," to present in chart form a means of obtaining quickly and easily the number of winder sets of known size from a reel of given diameter.	C. H. Reagan
4052	Industrial Engineering and Materials Handling	"Storage Aisle Width Requirements," to compile and present in chart and data form, storage aisle width requirements for paper and paper products warehousing.	C. E. Howard
4053	Industrial Engineering and Materials Handling	"Chart and Diagram Covering Normal Work Place," to prepare and present a chart and diagram covering normal work place for industrial workers, based on generally accepted standards.	C. H. Reagan
4054	Industrial Engineering and Materials Handling	"Tractive Effort on Inclines and Ramps," to present in readily usable form data covering required tractive effort of common industrial vehicles on inclines and ramps under various conditions.	C. H. Reagan
4055	Industrial Engineering and Materials Handling	"Standard Performance for Trucks," to obtain and present useful data on standard performance of various industrial trucks.	C. E. Howard
4056	Fluid Mechanics	"Head of Stock Behind Slice," revision of Data Sheet No. 3-3A.	G. L. Calehuff
4057	Fluid Mechanics	"Flow Characteristics of Dilute Fiber Suspensions: Turbulent Transfer Mechanics with Neutrally Buoyant Suspensions (TAPPI Project Appropriation CA-715-PA-192)," this is a continuation of studies on the flow of dilute fiber suspensions. The specific purpose of this work is to isolate and examine some of the key factors in relation to their effect in the flow of papermaking fiber suspensions (by key factors, we mean turbulent mixing and diffusion processes).	S. G. Mason
4058	Fluid Mechanics	"Study of High Speed Free Surface Flow Instabilities Associated with Fourdrinier Paper Machine Operation (TAPPI Project Appropriation CA-896-PA-191)," the purpose of the research grant is to sponsor research at the University of Michigan to develop an understanding of the causes of instabilities (i.e., uncontrolled disturbances) in the flow onto and over the Fourdrinier paper machine.	A. C. Spengos
4059	Corrosion	"Corrosion Resistance of Alloys in Pulp and Paper Mill Service," develop information on the corrosion resistance of metals and alloys in the various corrosion exposures in the pulp and paper mill installations.	R. C. Stamm
		TESTING DIVISION	
8025	Pulp Testing	T232 "Fiber Length of Pulp by Projection," complete revision.	J. d'A. Clark
8026	Pulp Testing	T234 "Coarseness of Pulp Fibers," complete revision.	J. d'A. Clark
8027	Optical Methods	"Procedure to Measure Whiteness of Pulp and Paper," specification of procedure and apparatus.	J. M. Patek

Corrugated Containers Division

Production Committee

The meeting was called to order by Chairman Loren Ashwood at 9:00 a.m., Feb. 21, 1961, in Parlor F, Commodore Hotel.

Attendance was recorded and the meeting followed the prepared agenda.

PROJECTS

Project No. 695—Adhesive Factors. Some conflict in the starch consumption figures was pointed out by Metin Hammarat. It was suggested that plants running all three major flutes should be used for this study. Mr. Buttery stated that a report of the study will be sent to each member of the committee. Any questions on the report should be referred to Ralph Buttery, who will then discuss them with Professor Germain in Winnipeg and submit the answers to the committee.

Our Whippet Jordan; the Paper Industry's "COMPACT"

It isn't THIS small, BUT . . .

. . . this rugged little producer does a BIG job at less cost to you. **Why use a large Jordan for a job that doesn't require it?**

This small refining specialist gives high speed production on all grades at a real saving of horsepower. Range, 10-50 tons; allowable speed, 500-1200 R.P.M.; H.P., 40-125.

Write or phone for full information.

NOBLE & WOOD

MACHINE COMPANY

Paper Mill Machinery

Hoosick Falls, New York, U.S.A.

NOBLE & WOOD ACCEPTS A NEW CHALLENGE ENTHUSIASTICALLY, LED BY A VIGOROUS NEW MANAGEMENT TEAM AND BACKED BY 67 YEARS OF SUCCESSFUL EXPERIENCE

Project No. 781—Errors. Mr. Hurrell, project leader, summarized the results of a meeting of his committee on this project. It was felt that further investigation would require some basic definitions and that an additional questionnaire would not be of general benefit. It was again pointed out that the clerical aspect of errors was very important and that most companies should direct more attention in this area for error reduction.

Project No. 786—Supervisory Training. Mr. Ashwood distributed lesson plans on company policy, quality control, and errors. Lesson plans on union contracts will be ready for distribution by the next meeting. Keith Provo and Loren Ashwood both reported using these lesson plans in their "supervisory training programs" with excellent results.

Project No. 791—Production Reports. Mr. Provo, project leader reported on the study of corrugator production with a data sheet showing the results of production figures submitted for two months production. Mr. Mendlin made the recommendation that at the next committee meeting the various companies, which showed up as efficient operations in the production reporting, have their representatives give short talks, describing their operations and pointing out the various reasons which they felt were mainly responsible for their good showing.

Additional studies for other equipment in printing and finishing was recommended. Mr. Provo will submit additional questionnaires for this.

A motion was made by Mr. Mendlin and seconded by Mr. Wilson, commending Messrs. Provo and Kaley on an excellent job done on the report submitted on corrugator production.

Mr. Mendlin reported on the Steering Committee Meeting.

Mr. Wilson reported on the fall meeting, to be held in San Francisco. Mr. Caploe will replace Mr. Kaley as moderator and Mr. Catzen will act as the other moderator.

Membership

Mr. Glass reported on committee size and active members. Questionnaires were sent out to all committee members to determine who desired to remain active. This procedure will be followed yearly to keep the list active.

New Projects

A discussion was held concerning new projects for investigation by the committee. The problems of controlled counts, standard procedures on overruns and shortages, employee training, standardization of terminology, waste control, relationship of pay for supervisors and production employees, control of matched sets, and counters were discussed to some extent.

Messrs. Mendlin and Glass both reported excellent results with a counter on the printer slotters, which uses sound waves on the same principle as the electric-eye counters use light waves. This counter is manufactured by the ARO Equipment Corp. of Bryan, Ohio. The unit which has proved itself in use on slotters is the 655–001, general purpose sonac counter.

A committee was appointed consisting of J. Simpson, chairman, and D. Adams, to make a study on controlling count or reducing shortages and overruns.

It was recommended that the TAPPI Steering Committee consider the problem of standardization of terminology in our industry.

On employee training, a discussion was held on the possibility of committee sponsored films being made on various phases of our operations in box shops, covering safety, need for quality, service to customers, demonstration of some skills or jobs, etc.

A suggestion by Mr. Hammarat was made that a committee be appointed to look over the various material already on hand. Short films on the various subjects not already covered could be made for training the employee. A committee consisting of Mr. Catzen, chairman, and Mr. Williams was appointed to review any material on hand and to make recommendations at the fall meeting, as to material suitable for training and training film needed.

On the question of relationship of pay for supervision and production employees, it was recommended that this study be turned over to the Industrial Engineering Committee.

The discussion on waste resulted in the naming of a committee with Frank Albert, chairman, and George Caploe, to study the problem of defining customers waste and the various methods of computing customers waste.

Organization

Chairman Ashwood appointed Messrs. Buttery and Provo as nominating committee to present a slate of officer candidates for 1962 at the fall meeting in San Francisco.

February 1962 Meeting

The Production Committee has responsibility for part of the technical program at New York in 1962. Mr. Ashwood appointed Mr. Comly and Mr. Simpson as moderators for that meeting.

Frank H. Glass, *Secretary*

Interlaboratory Evaluation of Test Methods*

New Tentative Testing Practice T 1200†

THE purpose of an interlaboratory study is to determine: (1) the variability in results caused by different personnel, equipment, and environment in following a prescribed method of test; (2) the consistency from material to material of this variability; and (3) the type of additional standardization needed, if any. The interlaboratory test may also include a comparative study of two or more alternative methods of test.

In order to achieve these objectives satisfactorily and efficiently, it is essential that sound statistical designs be employed in the planning of interlaboratory studies. This recommended practice gives the basic principles involved in the planning, and also in the analysis and interpretation of an interlaboratory study.

It has been written for a task group chairman who is responsible for the preparation or revision of a standard test method. It tells him what information he needs to provide to a statistician selected to help in planning the details of an interlaboratory study, or, if one is not available, enables him to plan the interlaboratory study himself.

OUTLINE OF RECOMMENDED PROCEDURE

Formulating the Problem

The objective of the task group should be clearly spelled out when the task group is established to study the problem at hand. This should be obtained by task group discussion, and approval obtained from the chairman of the parent committee (see Appendix I, Section A).

Preliminary Study Within One Laboratory

1. Investigate the different variables of the method, both experimentally and theoretically, including the following (see also Appendix I, Section B):

(a) Survey the literature and other sources of information.

(b) Determine how the result of a measurement may be affected by variations in critical instrument dimensions or critical steps in the procedure.

(c) Determine how the result may be affected by known variations in atmospheric conditions, unknown errors by different operators, and by any other variables that can be established.

2. Select what appears to be the optimum procedure. A choice between two procedures may be made using the "sensitivity criterion" (Appendix II).

3. Using papers with a wide range of values of the properties under test (and possibly also with wide ranges in other properties), make a comparative study with other methods for measuring the property. (See also Appendix I, Section B (d)) and Appendix II. Evaluate the results using the sensitivity criterion, when applicable, or an absolute standard.

4. Draft instructions for the method, after which observe, without comment, a laboratory technician perform a test according to this draft. If he has difficulty, revise the wording of the draft.

Study by Task Group

Prepare a clear statement of the type of information the task group expects to obtain from the interlaboratory study.

Based on the study within one laboratory, prepare a master plan for the interlaboratory study as will be described. Circulate the proposed plan to all members of the task group and other competent authorities (including the manufacturers of the instruments involved) for comment and criticism. Discuss the plan in an open committee meeting.

Select the materials to be used in the interlaboratory studies: (1) which will cover the applicable range of the property to be measured, and (2) which will represent all classes of materials to which the method will be applied.

Pilot Study

If the method is new or the procedure for an old method is greatly altered, a pilot study involving two or three materials and four or five laboratories should turn up any seriously misleading directions in the procedure. It is extremely important that the directions for performing the test be clear, unambiguous, and sufficiently comprehensive.

First Interlaboratory Study

For the first interlaboratory study use a minimum number of materials (six) to cover the range. Include all the laboratories that will participate in the main interlaboratory study and send to each a check list covering the requirements for each critical part of the apparatus or step of the method, to ensure that each requirement is complied with. This study is to train the participants in the method, clarify the procedure, eliminate laboraotries that cannot comply with the procedure because of nonstandard conditions or equipment, and, together with the main study, to give an indication of the constancy with time of laboratory results.

* This method was prepared by a joint ASTM–TAPPI task group in cooperation with members of ASTM D-6 Subcommittees, the TAPPI Paper and Paperboard Testing Committees, and the TAPPI Precision Committee.

† This method has been approved by the Standards Committee as a Recommended Practice. Criticisms are invited and should be sent to the Technical Secretary, TAPPI, 360 Lexington Ave., New York 17, N. Y.

Main Interlaboratory Study

For the main interlaboratory study follow the preceding section, only use a maximum number of materials. Include only those laboratories which participated in the first laboratory study and can meet the standard requirements, unless there are too few such laboratories.

Decision on Standardization

This will be based on the results of the main interlaboratory study (as will be discussed). The decision could be (1) abandon the method, (2) use the method as is, (3) rewrite the procedure to eliminate some of the variability, or (4) provide one or two standard samples for "calibrating" the method.

INTERLABORATORY STUDIES

Design

Master Plan. Use the plan agreed upon by the task group after careful discussion. This plan should include the following:

1. Instructions on care of round-robin specimens and what to do in case the specimens or results are lost.
2. Instructions on the adjustment and calibration of test apparatus.
3. Instructions on the order of testing the specimens.
4. Instructions on recording the results on the report form.
5. Detailed instructions for performing the test.

Every precaution should be taken to see that every laboratory follows all the instructions in the master plan, such as including a check list as previously mentioned.

Materials. Use a minimum of six materials for a single-scale instrument, and a minimum of five materials per scale for a multiple-scale instrument, the five or six materials covering the useful range of the scale. The more widely different the types of materials to be included in the study, the more materials per scale will be required.

Laboratories. Use as many laboratories as practicable, not less than 10, and preferably 20 or 30, subject to the amount of work involved in preparing samples for distribution to the participating laboratories, and the increase in sampling variability due to the larger quantity of material required. If fewer than 10 laboratories are prepared to use the method, include all the laboratories that will do so. Obtain assurance from each participating laboratory that it is properly equipped to follow all the details of the procedure as written and is willing to assign the work to competent personnel.

Replications. Use as many replications as are practicable (at least four), consistent with economic considerations of time and material and statistical considerations as to the homogeneity of each material. However, it is better to have a large number of materials than to have a large number of replications per sample of material.

Note:—Ideally each replicate test should be a completely independent determination. No steps in the determination should be made simultaneously on replicates.

Allocations of Specimens. Allocate and distribute the specimens from a single place. Prepare enough samples from each material to provide the required test material for all participating laboratories and a sufficient number of additional samples for replacement of those lost or spoiled. Label each sample by means of a code symbol and record them for future reference. Completely randomize the samples of a particular test material before dividing them into the required number of groups for assignment to the laboratories.

Effect of Aging of Samples. If the samples are of such a nature that their properties may change noticeably in the course of days or a few weeks, the timing of the tests should be coordinated among the participating laboratories, so that the effect of aging is not confounded with the differences among laboratories.

Report Form and Questionnaire. Supply each participating laboratory with a form for reporting the data to insure that all pertinent data and information are reported in a uniform manner by all participants. In addition to providing space for reporting the measurements and the usual information, such as relative humidity, temperature, and type of instrument, the report should also include a questionnaire or the check list, the answers to which will tell whether all the critical steps in the procedure have been followed correctly.

Analysis of Data

The analysis of the data is made in accordance with the "linear model," which assumes that systematic differences exist between sets of measurements made by different laboratories, and that these systematic differences are linear functions of the magnitude of the measurements.

Consider a particular laboratory and suppose that for each material the average value obtained by this laboratory is plotted against the average obtained for this material by all the laboratories combined. Then, according to the linear model, the points corresponding to the various materials will lie on a straight line except for random errors. The same applies for all the other laboratories. Thus the results of the round-robin may be represented by a set of straight lines with

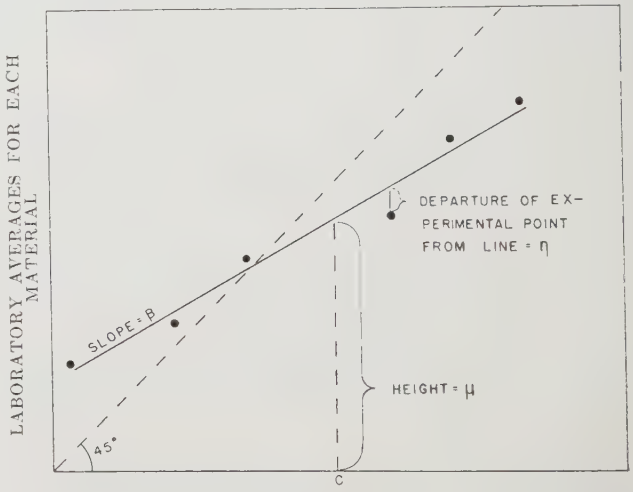

LABORATORY AVERAGES FOR EACH MATERIAL
SLOPE = β
DEPARTURE OF EXPERIMENTAL POINT FROM LINE = η
HEIGHT = μ
45°
c
AVERAGES OVER ALL LABORATORIES FOR EACH MATERIAL

Fig. 1. Linear model

c = average of all materials and all laboratories. η may be divided into two parts: that which can be accounted for by replication error (ϵ) and that due to differential response of that laboratory to that material (λ).

common abscissas. Each laboratory is characterized by the slope β and the location parameter μ of its straight line, and the departure η of each experimental point from the line (Fig. 1).

The departure of an experimental point from its line (within-laboratory variability) may be divided into two parts: that explainable by replication error, ϵ, and that which is not, λ. The latter may be thought of as the differential response of that laboratory either to some interfering property of that material (such as permeability, or response to relative humidity) or as a result of instrument scale error. Hence, the total error of the test method may be considered to be composed of two within-laboratory components, λ and ϵ, and two between-laboratory components, β and μ. Since the latter may be correlated, this must be taken into account in the analysis. If the data are transformed so as to give a within-laboratory variance, which is approximately independent of the magnitude of the measured quantity, the total variance, for any value of z in the transformed scale, is given by the following equation:

Total variability	=		within-laboratory variability			+	between-laboratory variability	
	=	random interaction*	+	replication		+	slope*	+ height
$V(z)$	=	$V(\lambda)$	+	$V(\epsilon)$		+	$(z - \bar{x})^2 V(\delta)$	$+ [\alpha(z - \bar{x}) + 1]^2 V(\mu)$

In this equation $V(\delta)$ is the part of the variability of β that is not related to μ, and $\bar{x}$ is the average value of μ.

For additional information see references cited.

Details of Computation

The following stepwise analysis of the data need not be understood by the task group chairman. It is presented for completeness and to aid the statistician in following the details of the computation.

Table I. Interlaboratory Study Involving *a* Laboratories, *b* Materials, and *n* Replications

	Materials					
	No. 1	*No. 2*	*No. 3*	... *j* ...		*b*
Laboratories						
No. 1	...	...	...	...		...
	...	.	...	...		
	...	..	...	...		..
No. 2	...	...	...	...		...
	...	..	...	..		.
	...	.	..	...		...
No. 3	..	..	...	...		..
	..	.	...	...		...
	...	..	.	..		...
...						
				i, j Cell		
i	...	...	...	...		...
	...	.	...	... }	*n*	...
	.	.	..	...		...
...						
a	...	..	..	...		...
	...	..	...	...		...
	..	..	.	.		..
Ave.	...	..	...	...		...

* In accordance with the linear model the classical laboratory-material interaction is broken into two parts: a systematic effect characterized by differences in slopes and a random effect characterized by V(λ).

Table II. Notation for Transformed Data[a]

	Materials						Average
	No. 1	*No. 2*	*No. 3*	... *j*	...	*b*	
Laboratories							
No. 1							
No. 2							
No. 3							
...							
i				z_{ij}			μ_i
...							
a							
Ave.				x_j			$\bar{x}$

[a] z_{ij} = cell average μ_i = row average, x_j = column average, and $\bar{x}$ = grand average.

Step 1. Scale Transformation. Arrange the data as in Table I. Compute, for each cell of Table I, its average and its standard deviation. Denote the average of the i,jth cell by y_{ij} and its standard deviation by s_{ij}. Then for each material (that is, column) compute the average $\bar{y}_j$ of the cell averages y_{ij} and the average $\bar{s}_j$ of the standard deviations s_{ij}. Prepare a graph plotting the standard deviation $\bar{s}_j$ *versus* the average $\bar{y}_j$. If the standard deviations appear to increase (or decrease) with increasing values of $\bar{y}_j$, fit a straight line to the points. Determine the intercept A and the slope B of this straight line, in accordance with the equation:

$$\bar{s}_j = A + B\bar{y}_j \qquad (1)$$

Since this equation is an approximation, A and B may be determined from a visual fit or a standard least squares technique.† The scale transformation to be used is then:

$$z = K \log (A + By) - C \qquad (2)$$

where K and C are arbitrary constants, the values of which are chosen on the basis of convenience. Theoretically, the transformation should be applied to each of the n observations in each cell. In most cases, however, it is sufficient to apply the transformation to the cell averages. Table II shows schematically the transformed averages z_{ij} for each cell.

As a result of the transformation, the standard deviation, and therefore also the variance, is constant for all cells in Table II. The variance is given by the following equation:

$$V(\epsilon) = \left(\frac{KB}{2.3}\right)^2 \qquad (3)$$

If the slope B is not appreciably different from zero, no transformation of scale is required, and all subsequent steps are carried out on the original cell averages. In this case, $y = z$ and $V(\epsilon)$ is the square of the average of all $\bar{s}_j$.

Step 2. Determination of μ_i, β_i, and $V_i(\eta)$. First

† Those who are acquainted with statistical theory will note that the standard error of $\bar{s}_j$ is not constant and will make the appropriate modification in the least squares procedure. However, these refinements will rarely have an appreciable effect in the outcome of the analysis.

compute the row and column averages in Table II as follows:

$$\mu_i = \frac{1}{b}\sum_j z_{ij} \tag{4}$$

$$x_j = \frac{1}{a}\sum_i z_{ij} \text{ and } \bar{x} = \frac{1}{b}\sum_j x_j \tag{5}$$

Then compute the quantities below in the indicated order:

$$X = \sum_j x_j^2 - b\bar{x}^2 \tag{6}$$

$$Z_i = \sum_j z_{ij}^2 - b\mu_i^2 \tag{7}$$

$$P_i = \sum_j x_j z_{ij} - b\mu_i \bar{x} \tag{8}$$

$$\beta_i = \frac{P_i}{X} \tag{9}$$

$$V_i(\eta) = \frac{a}{a-1}\cdot\frac{1}{b-2}\left[Z_i - \frac{P_i^2}{X}\right] \tag{10}$$

The last formula constitutes a slight departure from the ordinary calculations in linear regression: the correction factor $a/(a-1)$ is due to the fact that the errors of z_{ij} and x_j are slightly correlated.

Step 3. Determination of $V(\eta)$ and $V(\lambda)$. To obtain $V(\eta)$, average all values of $V_i(\eta)$. $V(\lambda)$ is given by the following equation:

$$V(\lambda) = V(\eta) - (1/n)V(\epsilon) \tag{11}$$

where $V(\epsilon)$ is given by equation (3). Should the estimate of $V(\eta)$ be less than that of $(1/n)\ V(\epsilon)$, then $V(\lambda)$ must be considered to equal zero.

Table III. Analysis of Variance

Sources of variation	*Degrees of freedom*	*Sums of squares*	*Mean squares*
Laboratories	$a-1$	S_L	M_L
Materials	$b-1$	S_M	M_M
Interaction (laboratory × material)	$(a-1)\times(b-1)$	S_{LM}	M_{LM}

Step 4. Determination of Variances. Table III is constructed in the usual manner, using the transformed cell averages. Note that $S_M = aX$ where X is given by equation 6, and that

$$S_{LM} = \left(\sum_j Z_i\right) - S_M = \left(\sum_i Z_i\right) - aX \tag{12}$$

The variances $V(\mu)$ and $V(\beta)$ may now be obtained from the previously obtained value of $V(\eta)$ and from Table III.

$$V(\mu) = \frac{M_L - V(\eta)}{b} \tag{13}$$

$$V(\beta) = \frac{a[M_{LM} - V(\eta)]}{M_M - V(\eta)} \tag{14}$$

If either of these equations yields a negative value, the corresponding variance is taken to be zero.

The quantity $V(\delta)$ is obtained from the following equation:

$$V(\delta) = V(\beta) - \alpha^2 V(\mu) \tag{15}$$

where $V(\beta)$ is given by equation 14 and α by the equation:

$$\alpha = \frac{ab\left[\sum_i \mu_i P_i - \bar{x}\sum_i P_i\right]}{S_L S_M} \tag{16}$$

If equation 15 yields a negative value, set $V(\delta) = 0$.

Step 5. Breakdown of Total Variance. The total variance of z, for any value of z, is given by the equation:

$$V(z) = V(\epsilon) + V(\lambda) + [1 + \alpha(z-\bar{x})]^2 V(\mu) + (z-\bar{x})^2 V(\delta) \tag{17}$$

From this equation we derive, by dividing by $V(z)$, the breakdown of the total variance into its fractional parts:

$$1 = \frac{V(\epsilon)}{V(z)} + \frac{V(\lambda)}{V(z)} + \frac{[1+\alpha(z-\bar{x})]^2 V(\mu)}{V(z)} + \frac{(z-\bar{x})^2 V(\delta)}{V(z)} \tag{18}$$

Step 6. Conversion to Original Scale. The total variance of z is converted back to the y-scale by means of the equation:

$$V(y) = \left[\frac{2.30}{K}\left(\frac{A}{B}+y\right)\right]^2 V(z) \tag{19}$$

It is generally desirable to add two more columns to the table showing the breakdown of $V(z)$: a column of the selected values for which the breakdown was made and a column of σ_y, obtained by taking the square root of equation 19.

Interpretation of the Analysis

The points of major interest are the relative importance of the various sources of error, the steps required to improve precision, if necessary, and the need for standard samples. For these purposes, the following procedure is recommended:

(*a*) Compare $V(\lambda)$ and $V(\epsilon)$: the relation between these two quantities will reveal how much can be expected from mere replication of measurements. If $V(\lambda)$ is large with respect to $V(\epsilon)$, replication is generally a waste of time. Even if $V(\lambda)$ is smaller than $V(\epsilon)$, replication is useful only to the point of making $V(\epsilon)/n$ small with respect to $V(\lambda)$.

(*b*) Study the table of values of β and μ for the various laboratories. Occasionally, a single laboratory (or a small group of laboratories) is discrepant in one or both these parameters, while all others are in close agreement. An investigation of the causes of such discrepancies is then indicated, and the analysis of variance carried out by omitting the discrepant laboratory (or laboratories) may be more meaningful than that based on its inclusion.

(*c*) Compare the total between-laboratory variability for various values of z with the within-laboratory variability, keeping in mind that the effect of $V(\epsilon)$ will depend on the number of replications that is called for by the standard method.

If the total between-laboratory variability is small compared with the within-laboratory variability throughout the table, all of the laboratories are essentially in agreement, and only refinement of the procedure to reduce $V(\lambda)$ can improve the precision of the method.

If $V(\lambda)$ is so large that the method is not sufficiently precise to be useful, the possible cause of a large $V(\lambda)$ should be investigated. Perhaps types of materials were included in the round robin for which the method

was not designed. Perhaps the method as written fails to call for the control of important interfering conditions or fails to correct for significant interfering properties.

If the between-laboratory variability is not negligible, examine its two terms separately. If the term in $V(\delta)$ may be neglected, the lines will form a simple pattern: they will either converge to a point (or a small region) or be, for all practical purposes, parallel. In either case, the calibration of the method at a single point other than the point of convergence will suffice to obtain the maximum possible agreement among the laboratories. On the other hand, if the term in $V(\delta)$ becomes appreciable anywhere in the table, the lines for the different laboratories will tend to criss-cross at random, and the method will require calibration at two points. There is one exception: if the term in $V(\mu)$ is negligible but not the term in $V(\delta)$, the lines happen to converge at the centroid, and calibration will be required at a single point as far away from the centroid as is practicable.

In general, the term in $V(\mu)$ will not be negligible throughout the table. If the variation in this term is small, the place of the required calibration point or points in the range of the measured quantity is immaterial, except that when two points are required, they should be located as far apart as practicable. If the variation in the $V(\mu)$ term is appreciable, the lines will partially or completely converge, and the calibration point or points should be located to avoid the area of convergence.

In summary, if between-laboratory variability is greater than within-laboratory variability and greater than can be tolerated for practical application of the method, the method must have better standardization. This can be done by using one or two standard samples to calibrate the method at appropriately chosen values of the measured quantity.

ADDITIONAL INFORMATION

This method is essentially the same as ASTM method D 1749–60 T which was issued in 1960 as a Tentative Recommended Practice.

APPENDIX

I. Technique of Evaluating an Instrument or a Test Method Within One Laboratory (1)

A. *Possible Objectives of Evaluation of a Method:*

(*a*) To determine whether the method actually measures the property intended, and how accurately.

(*b*) To determine whether test results correlate with results obtained by other commonly used methods to measure that property.

(*c*) To determine the limits and fields within which the method has value.

(*d*) To determine if certain defined faults of the method can and should be corrected.

B. *First Steps in Evaluation of Method:*

(*a*) Establish a clear understanding of the definition and significance of the measured property. Reconcile or recognize divergent interpretations of the property.

(*b*) Compare with known reliable methods or standards if possible.

(*c*) Examine individual principles and features of the method for compatibility with intended use.

(*d*) If reliable methods or standards are not available and if individual features cannot be usefully examined, compare with subjective methods and with other available methods, and remember that correlation is useful only if good for small differences in the property being measured as well as for large differences.

Appendix II. The Sensitivity Criterion

A. (*a*) "Sensitivity is a measure of performance which is useful in the evaluation of methods of test. Its application can be pictured in terms of an analogy with a radio receiver. Thus, an instrument or test method receives a signal in the form of a sample, and noise in the form of experimental error. The method detects and amplifies the desired signal (component to be determined) and yields a message in the form of a measured value. Sensitivity, in terms of this analogy, is a measure that increases directly with amplification and inversely with noise (*2*).

(*b*) Consider two alternative methods, A and B, for the measurement of a property, Q. For example, Q may be smoothness and A and B may be Bekk seconds and Sheffield number, respectively. Since both A and B are a measure of Q, they are functionally related to Q and therefore also functionally related to each other. The curve in Fig. 2 represents such a relationship. As can be seen from the figure, determination of the sensitivity ratio requires a knowledge of the slope $\Delta A/\Delta B$ and of the standard deviations, σ_A and σ_B.

B. Steps in the comparison of two methods or procedures by means of the sensitivity criterion are as follows:

(*a*) Determine the relationship between the results of the two methods so that the slope of this relationship can be evaluated at each point (that is, at each level of the measured property). If no relationship can be found, the two methods are *not* measuring the same property, and the sensitivity cannot be used for comparing them.

(*b*) For each method, determine the standard deviation and how it varies with the magnitude of the measurement. Remember that in order to obtain a good estimate of standard deviation, considerable replication is required.

The nature of the standard deviation occurring in the definition of sensitivity depends on the nature of the comparison between the two methods. In a within-one-laboratory study, the interest is centered on the intrinsic merit of each instrument. Therefore, the

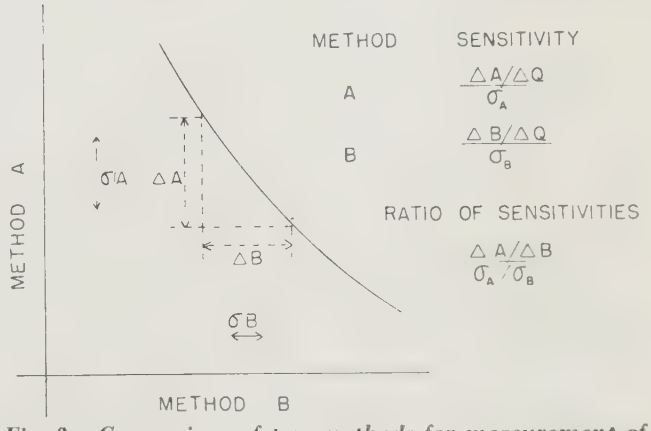
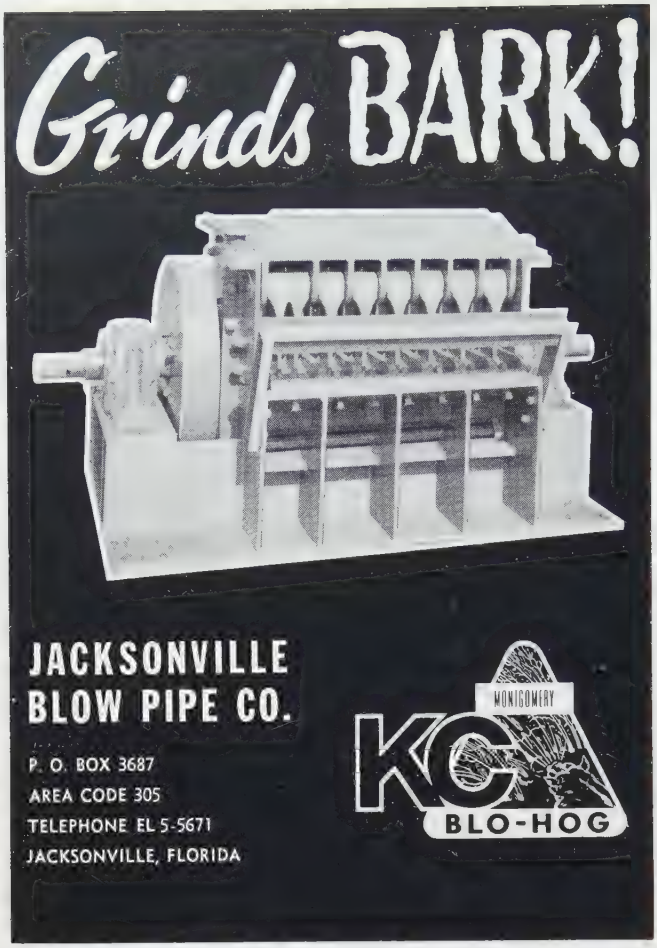


Fig. 2. Comparison of two methods for measurement of property Q



standard deviation used in the evaluation of sensitivity must be a measure of instrumental fluctuations only. Consequently, all errors that are not related to the instrument, such as variability in machine and cross-direction, sheet-to-sheet variability, and systematic operator biases, are eliminated. The remaining error is used in the estimation of the standard deviation.

If two methods are compared on an interlaboratory basis, as when deciding which shall be chosen as the standard method, the standard deviations to be used are the total standard deviations (or standard errors) obtained from the interlaboratory study.

(*c*) Compute the ratio of sensitivities in accordance with the formulas given in Fig. 2. Method *A* is more sensitive wherever the ratio is appreciably greater than unity.

LITERATURE CITED

1. Sears, G. R., Wink, W. A., and van den Akker, *Tappi* 35: 156A (Aug., 1952).
2. Stiehler, R. D. and Mandel, J., "Evaluation of Analytical Methods by the Sensitivity Criterion," *Anal. Chem.* 29: 17A (April, 1957).

REFERENCES

Mandel, John and Lashof, T. W., "The Interlaboratory Evaluation of Testing Methods," ASTM Bulletin No. 239: 53 (TP 133) (July, 1959).

Mandel, John, "The Measuring Process," *Technometrics* 1, No. 3: 251 (Aug., 1959).

Mandel, J. and Stiehler, R. D., "Sensitivity—A Criterion for the Comparison of Methods of Test," *J. Research, Nat. Bureau Standards* 53, No. 3: 155 (R.P. 2527 (Sept., 1954)).

Lashof, T. W., Mandel, John, and Worthington, V., "Use of the Sensitivity Criterion for the Comparison of the Bekk and Sheffield Smoothness Testers," *Tappi* 39: 532 (July, 1956).

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RC 301. Surface Wax in Paper

The object of this method is to determine the amount of surface wax in paper, by a scraping process.

Test Specimen

A measured area of waxed paper, approximately 6 by 6 in.

Apparatus

A laboratory balance, sensitive to a milligram. A sharp-edged instrument, such as a razor blade or edge of a microscope slide.

Procedure

1. Cut the paper sample approximately 6 by 6 in., and record the exact dimensions.
2. Weight the sample to the nearest milligram.
3. Lay the sample on a paper pad and scrape wax off the top side, continuing until all the wax appears to be removed. Avoid excessive removal of paper fibers.
4. Weight the sample.
5. Repeat the process on the other side of the sheet (if it is waxed on both sides.)
6. Calculate the amount of wax removed in pounds per ream ($24 \times 36 - 500$) on each side by the following formula:

$$\text{Pounds per ream} = \frac{g \times 952.4}{A}$$

where

g = weight of wax in grams.
A = area of test specimen in square inches.

RC 302. Rapid Determination of Wax in Paper

This is a simplified method suitable for the determination of wax impregnated paper by extraction.

The apparatus used in this determination is a weighing bottle and a beaker.

This method is intended as a rapid procedure for the determination of the wax content of paper. It is not intended for use with paraffin sized papers.

Procedure

A test specimen of approximately 0.25 g. is placed in a weighing bottle and covered with 1,1,1-trichloroethane for 1 min. The solvent is then poured off into a weighed beaker, and two more 1-min. extractions are performed. The combined portions of solvent are then evaporated over a steam bath, and the weight of the wax residue determined. The wax content is reported as a percentage of the original weight of the waxed paper. Duplicate determination should agree within 0.3%.

RC 303. Porosity of Paper and Paper Board

The object of this method is to measure the porosity of paper and paper board by measuring the flow of air through a paper sample clamped in the porosity testing head.

Apparatus

Bendtsen smoothness and porosity tester.*

Procedure

1. Plug in the compressor and place the 75-mm. weight on the spindle of the regulator.
2. With air flowing to the porosity head, place the paper in position and gently lower the handle until the head contacts the paper, and further until the handle just reaches its stop.

RC 304. Hardness of Paper and Paperboard

The object of this method is to measure the hardness of paper and paperboard by measuring the flow of air across the narrow contact ring of a smoothness head resting on a paper sample under two different weights.

Apparatus

Bendtsen smoothness and porosity tester.*

Procedure

1. Plug in the compressor and place the 150 mm. weight, which should be kept in its box when not in use, on the spindle of the regulator.
2. When the spinning weight has reached a uniform speed, check the calibration with the standard capillary. Remove the hose leading to the porosity head from the column, replace it with the hose on the standard capillary, and switch the valve to this outlet. (*Note:* a daily check is sufficient.)
3. Clean the glass plate and smoothness head with the camel hair brush provided, place the paper sample on the plate, and with air flowing to the smoothness head, place it gently on the paper.
4. Record the first stable reading of the float. Its position may change slowly from the initial reading, so this reading should be taken without delay. This is the smoothness valve.
5. Gently place the compression weight on the smoothness head so that it is centered with its projection fitting into the depression of head, and again record the first stable reading. This is the compression value. The hardness is the ratio of the compression value to the smoothness value found in step 4. RC Method 305.

RC 305. Smoothness of Paper and Paperboard

The object of this method is to instantaneously measure the smoothness of paper and paperboard by measuring the flow of air across the narrow contact ring of a smoothness head resting on a paper sample.

Apparatus

Bendtsen smoothness and Porosity tester.*

Procedure

1. Plug in the compressor and place the 150 mm. weight,

* This instrument may be purchased from the Robbins Instrument Co., 110 W. 30th St., New York 1, N. Y.

which should be kept in its bow when not in use, on the spindle of the regulator.

2. When the spinning weight has reached a uniform speed, check the calibration with the standard capillary. Remove the hose leading to the porosity head from the column, replace it with the hose on the standard capillary, and switch the valve to this outlet. (*Note:* a daily check is sufficient.)

3. Clean the glass plate and smoothness head with the camel hair brush provided, place the paper sample on the plate, and with air flowing to the smoothness head, place it gently on the paper.

4. Record the first stable reading of the float. Its position may change slowly from the initial reading, so this reading should be taken without delay. This is the smoothness valve.

5. When finished testing, place the smoothness head and compression weight in their holders on the base, and remove the regulator weight from its spindle before turning off the compressor.

RC 306. Coating Pick Tests Using the Interfibre Bond Tester

A modification of the interfiber bond tests as performed on the Interfibre bond tester* allows this tester to be used as a pick tester, thereby enabling rapid, accurate pick determinations with very little or no training.

Procedure

1. Prepare a paper specimen 1 by 6 in. in length.

2. Prepare a wax paper mask (household grade 0.002 in. thick), 1 by 6 in. in length. Punch five $^1/_2$ in. diam. holes exactly 1 in. apart in this wax paper.

3. Place the five specimen lower anvils in position in the specimen preparation station.

4. Place the first layer of double side tape into place on the bases.

5. Lay the centerboard mask of wax paper over the tape centering the holes on each base.

6. Lay the specimen above the mask.

7. Lay the second layer of tape on these assembled specimens.

8. Assemble the angles in the strong back. With the angles in the proper position, lay assembly on the specimen preparation station and lock down with the thumb screws.

9. Using the lever, apply load to the specimen. (The load of 50, 100, 150, or 200 p.s.i. can be used; however, the operator will have to judge how deep into the body stock he wishes to penetrate and adjust his load accordingly.)

10. Operate the tester in accordance with the instructions provided by the manufacturer.

11. Report results in thousandths of a foot pound.

RC 307. Deinkability of Wastepaper

Tear 10 g. of a representative portion of the wastepaper into approximately 1-in. squares and add to a 600-ml. beaker containing 0.8 g. of NaOH in 250 ml. of hot water (about 190°F.).

Maintain temperature between 190°F. and a low boil for 10 min., transfer to a Waring Blendor, and stir at high speed for 30 sec. (Hold cover down firmly because of possible slop). After the first 10 sec. stirring, stop the stirrer, loosen any "hung" paper with glass rod and stir the additional 20 sec.

Drain the suspension on a 6 in. diam. 60 mesh sieve and wash as follows: (1) stir the pulp into 500 ml. of fresh cold water, using a stirring rod, and drain again on the sieve. (2) Repeat step (1) twice more.

Stir the washed pulp into 1000 ml. of fresh cold water, using a stirring rod, and proceed as follows:

1. Use 400 ml. of the pulp suspension to make a handsheet on Noble & Wood or equivalent sheet machine.

2. Drain 400 ml. of the pulp suspension on a 60-mesh sieve, transfer to a 150-ml. beaker and add 4 ml. of soda bleach (30 g. available chlorine per liter). Blend promptly by pouring the mixture slowly into a second beaker while stirring with a glass rod. Complete blending by pouring back into the first beaker while stirring. Cover beaker with watch glass and hold at 110°F. for 4 hr. Then make a handsheet of the bleached pulp, regardless of the presence of any unconsumed bleach.

3. The remaining 200 ml. of pulp suspension should be poured into a shallow black pan and inspected for visible nonfibrous particles and for bits of undefibered paper. These should be removed from the suspension and lined up on a clean blotter for inspection.

4. Observe both handsheets (wet and after drying) for general cleanliness by both reflected and transmitted light, inspecting with the aid of a low power magnifying glass like a thread counter. Observe the presence and nature of the bits of undefibered paper and of the nonfibrous particles taken from the shallow pan. Inspection should indicate whether there is wet-strength paper, coating fragments, inkballs, plastic or rubbery fragments, or other objectionable particles.

5. The appearance and brightness of the unbleached and bleached handsheets and the results of the inspection of the suspension in the shallow black pan should be a satisfactory basis for judging the deinkability of the wastepaper from the standpoint of the end use of the paper to be made from the deinked pulp.

Notes: If it is desired to substitute some other type of mixer for the Waring Blendor, it will first be necessary to prove that the method gives acceptance to samples known to be good, and rejection to samples known to be bad, when the other mixer is used. This can best be done by running selected papers in a Waring Blendor and in the desired mixer for comparative tests.

RC 308. Test for Interfiber Bond Using the Internal Bond Tester

This method is designed to determine the average force in thousandths of a foot pound required to separate a paper specimen, to determine the internal bonding strength of the paper specimen.

Apparatus

The testing apparatus shall be in accordance with the following requirements. It shall have:

1. A stationary anvil and separable platen which the pendulum shall strike at the same point for each test.

2. A pendulum of dual capacity, 0–0.250 and 0.100–0.500 ft. lb., and free to swing on a ball bearing or similar low friction type bearing. The range changing weights must be placed so as not to alter the center of gravity of the pendulum.

3. A means of holding the pendulum in a raised position with provision for instantaneous release.

4. A means of registering the maximum arc through which the pendulum swings when released.

5. A scale calibrated in thousandths of a foot pound on both ranges, with a reference point for calibrating the pointer in free swing to compensate for friction and windage.

6. A method of multiple specimen preparation which will allow for variations of specimen thicknesses, with optional clamping pressures on the specimen from 50 to 200 p.s.i. in 50 p.s.i. increments.

Discussion

A paper specimen bonded on both sides by a double sided adhesive paper tape which in turn is adhered to a metal test fixture is impacted by means of pendulum at about 90° from the release point parallel to the surface of the paper.

The impact results in delamination of the weakest point of

* Scott Testers, Inc.

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for
SMOOTHNESS . . . POROSITY . . . HARDNESS
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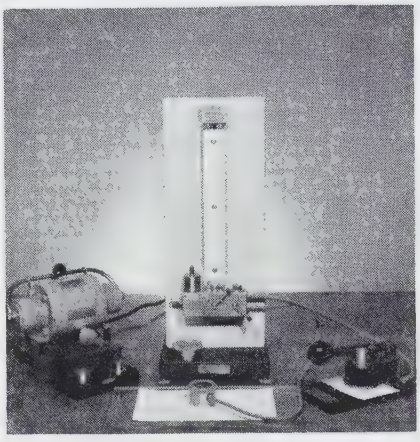

INSTANTANEOUS: No waiting—No scale adjustments.
ACCURATE: No oil or dust can enter instrument. Smoothness not effected by porosity.
VERSATILE: Completely self contained, portable, no special training required. Tests smoothness, porosity & printing hardness of paper & paperboard. Accessories available for paperboard & Hardboard.

Write for details of our 30 day free trial offer and our new time saving three tube tester

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the assemblage—namely the paper, between the lower and upper surfaces. The pointer of the tester indicates the energy required to rupture in thousandths of a foot pound of force.

Procedure

Level the instrument until the bubble of the integral spirit level is centered. The pendulum will then hang vertically coinciding with the vertical zero mark on the dial. The instrument must be calibrated before starting a series of tests. It must be recalibrated whenever the pendulum range weights are changed.

Specimens for the test shall be cut accurately 1 in. wide and 5 or more inches long. Care should be exercised to handle the specimens by the edges only. Specimens shall be conditioned according to TAPPI Standard T 402 before inserting into the specimen preparation station. Specimen preparation is performed as follows:

1. Load the tape dispenser with a 36-yd. roll of 1-in. Scotch brand type 400 double-faced pressure sensitive tape.
2. Select and set into position the desired pressure spacers in the bores of the specimen preparation station (use least pressure possible without peeling from angle or anvil). See Note 2.
3. Lay the bottom steel anvils in their respective position on the locating pins.
4. Pull out a strip of tape sufficient to go across the anvils and return. If any wrinkles appear on the tape, discard that section. Bring this tape down on the anvils between the guide pins. Place it carefully so that the tape exactly covers, but does not overlap the sides of the anvil. Place a strip of the paper to be tested on the tape in the exact position. Care should be exercised to avoid touching the paper with fingers except at the ends.

Swing tape hook up and over the tape at the end of the station. Bring the tape back over the hook and place it exactly on top of the specimen. If the tape is held under a slight tension, this is quite simple. Cut the doubled tape and specimen with scissors.

5. Install strong back loaded with aluminum angles and clamp with knurled thumbscrews.
6. Apply pressure by pulling forward the control lever on the right side of the machine for one or more seconds. Clamping too long will cause adhesive to penetrate specimens and test will be useless.
7. Release the pressure by returning the handle to its top position, unlock and remove the strongback carefully, leaving the aluminum sample angles adhered to the specimen sandwich. Separate each of the five specimens with the knife provided by inserting it between each pair of angles and cutting through the sandwich of paper and tape. Trim specimen making sure none overlaps the edges.
8. Swing the pendulum 90° to the right until it latches; swing pointer to this position also, until it touches pin on pendulum. Place specimen on the pedestal properly and secure assembly in place by tightening the knurled thumbscrew by hand.
9. Make the test by releasing the latch with the left hand index finger. Read the results and reset the pointer and pendulum. Remove the specimen holder and install the next one, etc.

Not less than five tests in each principal direction of the paper shall be made. Examine the shear of the samples carefully, checking for any indication of tape peeling (Note 1) from the specimen. If this is found, discard these results and increase the clamp pressure in 50-lb. increments until satisfactory results can be obtained. (Note 2).

Reporting

Report the results as a force of thousandths of a foot pound required to shear the specimen. The operator should exercise care so as not to allow any parallax to influence the values read from the scale. Results should be recorded to the nearest $^1/_2$ of a scale division. The average maximum and minimum results shall be reported for both the principal directions of the paper. Report results obtained from the test as internal bond strength of machine direction and internal bond strength of cross machine direction of the specimen. If the mean value of the lowest and highest readings differ from the average of all readings, excessively, test more specimens until there is agreement within agreeable limits.

Note 1—If one experiences excessive peeling of tape from the anvils, clean anvils with a suitable solvent or detergent to remove any oil film not compatible with the adhesive.

Note 2—Excessive pressure and/or time may result in forcing adhesive through the voids of the lighter paper stocks.

15th Alkaline Pulping Conference
November 1–3, 1961
Rice Hotel, Houston, Texas

Luncheon Speaker: Karl Bendetsen, President
Champion Paper and Fibre Co.
Hamilton, Ohio

Luncheon Speaker: Marvin Hurley, Executive Secretary
Houston Chamber of Commerce

Mill Tours:
Champion Paper & Fibre Co., Pasadena, Texas
East Texas Pulp and Paper Co., Evadale, Texas
Southland Paper Mills, Lufkin, Texas

Selection of Motors for Barking Drum Drives

Technical Information Sheet No. 8

The most important factors to be considered in selecting electric motors for the drive system of trunion-supported or chain-suspended barking drums are: (1) capacity required, (2) operating characteristics, and (3) motor location.

CAPACITY REQUIRED

The drive capacity selected will be influenced by the weight of the drum; the weight of wood in the drum (species, moisture content, and drum fill); drum speed; and whether or not the operation is of a wet or dry type.

As it is not possible to derive a nomograph which will include all these factors, and inasmuch as tests have shown that starting or break-away torques are normally greater than running torques, the use of the following systems are recommended for capacity determination:

Recommended Drive Capacities—Standard Drums

Standard drum sizes, ft.	*Type of Operation, hp.*	
	Dry	*Wet*
6 × 15	15	20
8 × 20	30	40
8 × 30	50	60
10 × 30	75	100
12 × 22.5	75	100
12 × 45	150	200
12 × 68	250	

Recommended Drive Capacities—Nonstandard Drums

Where nonstandard drum sizes are contemplated, the following formula will indicate the minimum horsepower required:

$$\text{Drive hp.} = D^2 LK \text{ (or } K_1\text{), ft.}$$

D = Drum diameter
L = Drum length, ft.
K = 0.023 (dry operation factor)
K_1 = 0.031 (wet operation factor)

Example of use: What is the recommended drive capacity for a barking drum having a 12 ft. diam., a length of 22.5 ft., and a dry operation?

$$\text{Drive hp.} = D^2 LK$$
$$= (12)^2 \cdot (22.5) \cdot (0.023)$$
$$= 75 \text{ (minimum)}$$

OPERATING CHARACTERISTICS

The following types of three-phase induction motors are recommended:

Wound Rotor Type

High starting torques; smooth acceleration; and no necessity for fluid couplings or mechanical clutches in the power train; are basic features of this type.

Starting torques may be increased by a maximum of 200% by the addition of proper secondary resistance, and although slip losses are greater than normal, starting current is materially reduced.

Due to these operating characteristics, the danger of mechanical damage to chains, gears, and shafting is greatly reduced. These factors favor the selection of the wound rotor-type motor even though its acquisition costs may currently be greater than that of the squirrel cage type.

Squirrel Cage Type

The normal starting torque of a squirrel cage motor is relatively low. Therefore, this type of motor should be used with a fluid coupling or a mechanical clutch, provided that the horsepower-speed characteristics of the coupling or clutch are coordinated with the motor. The coupling or clutch permits the motor to attain full speed very rapidly and allows use of the motor breakdown torque of 200% for smooth starting and acceleration. As indicated above, to obtain a successful drive the horsepower-speed characteristic of the coupling and clutch *must* be carefully coordinated with the motor. The cost of this combination is approximately the same as that of a wound rotor induction motor.

The use of a squirrel cage motor, without coupling or clutch, is not considered satisfactory due to the detrimental heating of the rotor during acceleration with frequent starting; and also due to the possible damage to chain, gears and shafting with abrupt starting and acceleration. Since the starting requirement is 150 to 175% of the full-load torque, high torque or proportionately oversized motors could be used. However, a squirrel cage induction has a definite limited duty-cycle and should only be applied with caution to machines that may start and stop frequently.

MOTOR LOCATION

Due to the unusually severe conditions normally found in barking drum operations, totally enclosed, fan cooled, or enclosed force-ventilated construction is recommended. However, where a minimum investment is dictated, semi-enclosed, splashproof, or weather protected construction may be considered in those situations where the power supply is no greater than 440 v.

ADDITIONAL FACTORS

In addition to the major items previously discussed, other design factors to be considered might be as follows:

1. Provision for externally enclosing the collector rings of wound rotor induction motors with a light weight and easily removable cover.

2. Provision of a lucite window, or equal, to allow quick inspection of rings and brushes.

3. Provision of filters for cleaning cooling air to totally or semi-enclosed motors. Where this feature is desired, a nonclogging type should be specified, and thermal protectors be included in the motor to prevent damage from overheating should a filter fail.

4. Provision of screens over any motor enclosure openings to exclude debris.

5. Provision of automatically energized space heaters to protect motor from condensation damage.

6. Provision of split-sleeve bearings or brackets to facilitate maintenance.

Prepared by: Electrical Engineering Committee (C.A. 746)

Guide for Use of Clad-Steel Plate

Technical Information Sheet No. 7

DEFINITION

A clad-steel plate is a composite plate made by uniformly and inseparably bonding a layer of some corrosion resistant metal to a carbon or low alloy steel backing plate. This may be done by hot rolling suitably prepared "sandwiches" to give the desired bond and plate thickness, or by vacuum furnace bonding of components already reduced to final thickness.

CLADDING METALS

A wide selection of cladding metals is available. Commonly used types are the 300 and 400 series stainless steels, nickel, and nickel based alloys.

BACKING METALS

A-285 grade C flange quality backing steel is usually furnished unless otherwise specified. Other steels such as A-201, A-202, and A212 are available when required.

BOND STRENGTH

Normal fabricating operations such as shearing, bending, flanging, forming, riveting, and welding can be performed without affecting the bond of clad-steel plates.

Clad plate manufactured under ASTM specifications A-263, A-264, or A-265 may be used in full thickness (minus corrosion allowance) for design of pressure vessels in accordance with the ASME Boiler Code. These specifications require a minimum bond shear strength of 20,000 p.s.i. for the cladding; commercial clad plate will usually possess bond strength twice this value.

CORROSION RESISTANCE

Clad-steel plate is generally applicable in all corrosion exposures where a solid plate of the same cladding material would be satisfactory. It must, however, be handled, formed, and welded so that the corrosion resistance of the cladding is not impaired.

The effective corrosion resistance of the cladding can be reduced by mechanical means, as well as by improper heating or welding. Scratches and gouges, particularly by carbon steel objects such as other plates or handling equipment, must be avoided. Such marks will not only reduce the protective thickness, but also leave smears of iron which may be attacked in service. If a crevice is formed and if other conditions favorable to concentration-cell corrosion are present, the attack can continue through the cladding. In the same way, a foreign particle imbedded in the clad surface not only may be corroded itself, but may lead to progressive corrosion of the cladding. Thus, it is important to guard against impressing or melting foreign material into the cladding during handling or welding.

HANDLING

In general, it is best to move clad plates with the cladding "up" to minimize scraping and scratching. Use of plate hooks for transferring is preferable. Where plate clamps are employed, use a piece of wood or stainless strip to prevent marring the cladding. Steel surfaces should not be piled directly against the cladding. Excessive accumulation of shop dirt in storage can cause fouling and rusting under the influence of moisture or be imbedded during subsequent forming. In forming operations, the rolls, brakes, and dies, as well as the clad surface, should be clean to prevent impressing scale or dirt into the surface.

For protection of the clad surface, paper covers may be used during storage and forming, or sprayed plastic or glued paper coatings may be applied by the manufacturer and left on through the forming operations. Plastic coatings are easily peeled off and the paper coatings are water-swabbed and stripped off. It is very important that such coatings are removed at least 2 in. from an edge to be welded or flame-cut to prevent a tightly adherent residue of burning. This applies also where a great deal of heat is to be applied on the backing side of thin plates.

FLAME CUTTING

Clad plates with up to 30% cladding thickness may be cut cleanly and easily with acetylene or natural gas and without powder injection. The technique involves cutting from the base steel side using large tip sizes and low oxygen pressures. The exact conditions depend upon total gage, percentage cladding, and the gas. Recommendations are available from the manufacturer or equipment supplier.

WELDING

Since clad is a composite of dissimilar metals, the electrodes and the welding procedure are selected to allow for this fact. Without going into detail, the following generalizations may be made:

1. Where an alloy is to be welded on the base steel, an electrode having higher alloy content or one specially

formulated for the purpose is used to compensate for dilution.

2. Clad plate $^3/_8$ in. and under is usually welded entirely with an alloy electrode, while plate over $^3/_8$ in. is usually welded by a combination of alloy and steel welds. Where a combination weld is used, the edge is prepared so that the steel side is welded first without picking up alloy from the cladding. The joint is then back gouged and the proper alloy electrode applied on the carbon steel. Dilution of carbon steel welds by the high alloys can result in brittle, or even cracked welds.

3. Superficial repairs or attachments of similar metals may be made with electrodes matching or compatible with the cladding metals and service. Attachments welded to the cladding will not cause bond loosening.

4. In preparing joints, welding, and conditioning, particularly for thin plates, care is taken to maintain the cladding thickness and continuity. Avoid weld undercuts and indiscriminate grinding.

Specific recommendations for weldnig clad are available from the manufacturer.

In welding clad plates, just as for homogeneous plates, arc strikes with electrodes or electrode holders should be avoided. Even arc strikes with a similar electrode may leave pores and fissures which under some conditions will result in progressive corrosion. Insulated electrode holders should be used. Avoid splatter of high alloy surfaces with carbon steel weld metal.

HEATING

Some of the 300 series stainless steels become sensitized when subjected to welding or hot forming operations and are therefore susceptible to corrosion by certain media. The columbium or titanium stabilized types, or the extra low carbon varieties, are more resistant to sensitization and consequently provide greater resistance to attack by those media.

PASSIVATION

Stainless steel vessels are sometimes cleaned with dilute nitric acid prior to installation. This treatment is chiefly valuable in that it may remove minor surface contamination or reveal gross defects. It is not considered a necessary treatment for the majority of applications where the alloy has been properly handled during fabrication.

Prepared by: Corrosion Committee (C.A. 810).

5 WAYS STONE & WEBSTER SERVES THE PAPER INDUSTRY

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LOCAL SECTION ACTIVITIES

Local Section Meeting Calendar

October, 1961

3 Chicago Section, Chicago Bar Association, 29 So. La Salle St., Chicago, Ill.

4 Western District, Empire State Section, Apple Grove Inn, Medina, N. Y. Visit to mill of U. S. Gypsum Co., Oakfield, N. Y.

5 Lake Erie Section, Cleveland Engineering Society Building, 3100 Chester Ave., Cleveland, Ohio.

6 Kalamazoo Valley Section, Hotel Harris, Kalamazoo, Mich. Joint dinner meeting with the Sixth Deinking Conference. J. D. Dailey, Sr., vice-president, Allied Paper Corp., speaker.

6–7 Pioneer Valley District, New England Section, Equinox House, Manchester in the Mountains, Vt. Joint meeting with Connecticut Valley PIMA.

6 Central District, Empire State Section, University Club, 431 E. Fayette St., Syracuse, N. Y. "A Review of the Principles of Continuous Cooking and a Description of the Latest Developments and Trends," by O. A. Laakso and Norman Baldwin, Kamyr, Inc., Hudson Falls, N. Y.

10 Metropolitan District, Empire State Section, Stouffers, 100 E. 42nd St., New York, N. Y.

12 Ohio Section, Visit to Research Facilities of Mead Corp., Chillicothe, Ohio. Research Seminar.

12 Northern District, Empire State Section. Mill visit to be announced.

17 Indiana District, Ohio Section, Terre Haute, Ind. Visit to mill of Weston Paper & Mfg. Co.

17 Lake States Section, Wausau Club, Wausau, Wis. Visit to plants of D. J. Murray Mfg. Co. and Wausau Iron Works.

19 Eastern District, Empire State Section, Queensbury Hotel, Glens Falls, N. Y.

20–21 Maine-New Hampshire Section, Mountain View Inn, Whitefield, N. H. Visit to mill of Brown Co., Berlin, N. H. Technical program devoted to high speed, high-temperature continuous cooking of hardwood and softwood.

26 Delaware Valley Section, Manufacturers Country Club, Oreland, Pa. Panel discussion on optical properties of paper.

November, 1961

1 Western District, Empire State Section, Crown and Anchor Restaurant, 2901 Pine Ave., Niagara Falls, N. Y. "Polyvinyl Adhesive and Coatings."

2 Kalamazoo Valley Section, Inman's Restaurant, Galesburg, Mich. "Sales-Technical Relationships," by H. W. Parnass, sales manager, Watervliet Paper Co., Div. Hammermill Paper Co., Watervliet, Mich.

2 Lake Erie, Cleveland Engineering Society, 3100 Chester Ave., Cleveland, Ohio. "Plastics in Packaging."

3 Central District, Empire State Section, Trinkaus Manor, Oriskany, N. Y. Visit to papermakers felt mill of H. Waterbury & Sons, at 2 p.m.

7 Chicago Section, Chicago Bar Association, 29 So. LaSalle St., Chicago, Ill. "Adhesives for the Corrugated Container Industry."

9 Northern District, Empire State Section, Woodruff Hotel, Watertown, N. Y. Executives' night.

11 Golden Gate Section, Visit to plant of IBM in San Jose, Calif.

14 Bay District, New England Section, Center Building, Arthur D. Little, Inc., Concord Turnpike (Route 2) Cambridge, Mass. "Isolating Paper Odors," by David A. Kendall, Arthur D. Little, Inc., Food and Flavor Section. Visit to laboratories, beginning about 5:30 p.m., will precede the dinner.

14 Pacific Section, Longview, Wash. "Paper Making and Screening."

14 Metropolitan District, Empire State Section, Stouffer's, 100 E. 42nd St., New York, N. Y.

14 Ohio Section, Manchester Hotel, Middletown, Ohio. "Modern Fine Paper Machines." Joint meeting with Miami Valley PIMA.

15 Lake States Section, Appleton Elks Club, Appleton, Wis. "Encapsilated Coatings."

16 Delaware Valley Section, Manufacturers Country Club, Oreland, Pa. "Drying of Paper."

16 Eastern District, Empire State Section, Queensbury Hotel, Glens Falls, N. Y.

17–18 Southeastern Section, Biltmore Hotel, Atlanta, Ga.

21 Berkshire District, New England Section, Country Club of Pittsfield (Mass.).

21 Indiana District, Ohio Section, Marott Hotel, Indianapolis, Ind.

30 St. Louis Section, Le Chateau, 10405 Clayton Rd., St. Louis, Mo.

30 Lake Erie Section, tour of plant of General Electric Co., Nela Park, Cleveland, Ohio.

December, 1961

1–2 Gulf Coast Section, Battle House, Mobile, Ala.

1 Central District, Empire State Section, University Club, 431 Fayette St., Syracuse, N. Y. "Engineering and Design."

1 Virginia-Carolina Section, Jefferson Hotel, Richmond, Va. Tour of a converting plant in afternoon.

4 Pioneer Valley District, New England Section, Storrowtown Tavern, West Springfield, Mass.

5 Chicago Section, Chicago Bar Association, 29 So. LaSalle St., Chicago, Ill. "Gloss Printing."

6 Western District, Empire State Section, Crown and Anchor Restaurant, 2901 Pine Ave., Niagara Falls, N. Y. "Black-Clawson Verti-Forma."

7 Kalamazoo Valley Section, Hotel Harris, Kalamazoo, Mich. "Pigment Coating," Afternoon-evening sessions.

12 Metropolitan District, Empire State Section, Stouffers, 100 E. 42nd St., New York, N. Y.

14 Ohio Section, Manchester Hotel, Middletown, Ohio. "The Potential of Mechanical and Chemical Pulps," by Renata Marton, Empire State Paper Research Association, Syracuse, N. Y.

14 Eastern District, Empire State Section, Queensbury Hotel, Glens Falls, N. Y.

14 Northern District, Empire State Section, Hotel Woodruff, Watertown, N. Y. "High Density Bleaching of Groundwood," by A. D. Armstrong, St. Regis Paper Co., Deferiet, N. Y.

19 Indiana District, Ohio Section, Marott Hotel, Indianapolis, Ind.

January, 1962

5 Central District, Empire State Section, University Club, 431 Fayette St., Syracuse, N. Y. Panel meeting. "On Machine Clay Coated Board."

8 Bay District, New England Section, Armand Restaurant, Framingham, Mass.

9 Lake States Section, Green Bay Elks Club, Green Bay, Wis. "Waste Wood Utilization." Visit to mill of Green Bay Paper & Pulp Co., Green Bay, Wis.

9 Metropolitan District, Empire State Section, Stouffer's, 100 E. 42nd St., New York, N. Y.

9 Chicago Section, Chicago Bar Association, 29 So. La Salle St., Chicago, Ill. "Fine Papers."

9 Ohio Section, Manchester Hotel, Middletown, Ohio. "Coating Processes and Technology," by George Booth, Dilts Machine Co., Fulton, N. Y.

11 Northern District, Empire State Section, Hotel Woodruff, Watertown, N. Y. Students' night.

12–13 Gulf Coast Section, Tuscaloosa, Ala.

16 Berkshire District, New England Section, Country Club of Pittsfield (Mass.).

16 Pacific Section, Bellingham, Wash. "Engineering."

16 Indiana District, Ohio Section, Marott Hotel, Indianapolis, Ind.

16 Golden Gate Section, San Francisco, Calif. Talk by W. F. Linke, American Cyanamid Co.

18 Eastern District, Empire State Section, Queensbury Hotel, Glens Falls, N. Y.

18 Kalamazoo Valley Section, Hotel Harris, Kalamazoo, Mich. Papermakers Get-Together.

19 Southeastern Section, DeSoto Hotel, Savannah, Ga.

February, 1962

1 Lake Erie, Cleveland Engineering Society, 3100 Chester Ave., Cleveland, Ohio. "Industrial Psychology."

2 Central District, Empire State Section, University Club, 431 E. Fayette St., Syracuse, N. Y. "Electron Photomicroscopy of Wood and Wood Fibers."

6 Chicago Section, Chicago Bar Association, 29 So. La Salle St., Chicago, Ill. "Testing Instruments Used for Boxboard and Paper," by George Maltenfort, Container Corp. of America, Chicago, Ill.

7 Western District, Empire State Section, Crown and Anchor Restaurant, Niagara Falls, N. Y. "Plastics in the Paper Industry."

8 Ohio Section, Manchester Hotel, Middletown, Ohio. "Computer Control for the Paper Industry," by Charles Carroll and David Kirk, I.B.M. Co.

8 Northern District, Empire State Section, Hotel Woodruff, Watertown, N. Y. "Headbox Hydraulics."

13 Indiana District, Ohio Section, Marott Hotel, Indianapolis, Ind.

15 Eastern District, Empire State Section, Queensbury Hotel, Glens Falls, N. Y.

March, 1962

1 Kalamazoo Valley Section, Hotel Harris, Kalamazoo, Mich. "Developments in the Graphic Arts."

1 Lake Erie, Plant tour.

2 Central District, Empire State Section, University Club, 431 Fayette St., Syracuse, N. Y. "Instrumentation of Paper Machines," by L. L. Thompson, I.B.M. Research Center, Yorktown Heights, N. Y.

6 Chicago Section, Chicago Bar Association, 29 So. LaSalle St., Chicago, Ill. "Foamed Plastics as a Packaging Medium."

7 Western District, Empire State Section, Crown and Anchor Restaurant, Niagara Falls, N. Y. "Stream Improvement."

8 Northern District, Empire State Section, Hotel Woodruff, Watertown, N. Y. Junior Award Papers.

12 Bay District, New England Section, Armands Restaurant, Framingham, Mass.

13 Ohio Section, Student Center, Miami University, Oxford, Ohio. "New Fibers from Wood," by Herman Mark, Polymer Research Institute, Brooklyn, N. Y. Joint meeting with Indiana District and students and faculty of Miami University Paper School.

13 Metropolitan District, Empire State Section, Stouffer's, 100 E. 42nd St., New York, N. Y.

13 Lake States Section, Mead Hotel, Wisconsin Rapids, Wis. Panel discussion on "Paper Finishing Operations." Visit to mill of Nekoosa Edwards Paper Co., Port Edwards, Wis.

15 Eastern District, Empire State Section, Queensbury Hotel, Glens Falls, N. Y.

16–17 Southeastern Section, Wilmington, N. C. Visit to mill of Riegel Paper Corp., Acme, N. C.

20 Pacific Section, Camas, Wash.

20 Indiana District, Ohio Section, Marott Hotel, Indianapolis, Ind.

20 Berkshire District, New England Section, Wendell Sherwood Hotel, Pittsfield, Mass.

20 Golden Gate Section, San Francisco, Calif. Panel discussion on "Functional Coatings."

22 Delaware Valley Section, Manufacturers Country Club, Oreland, Pa. E. G. Albert Award papers.

22 24 Gulf Coast Section, San Carlos Hotel, Pensacola, Fla.

23 Virginia-Carolina Section, John Marshall Hotel, Richmond, Va.

April, 1962

2 Pioneer Valley District, New England Section, Storrowtown Tavern, W. Springfield, Mass.

3 Chicago Section, Chicago Bar Association, 29 So. La Salle St., Chicago, Ill. "Preprinting of Corrugating Liner."

4 Western District, Empire State Section, Crown and Anchor Restaurant, Niagara Falls, N. Y. Visit to plant of Hazel Atlas Glass Co.

5 Kalamazoo Valley Section, Hotel Harris, Kalamazoo, Mich. "Chemi-Mechanical Pulping."

5 Lake Erie Section, Cleveland Engineering Society, 3100 Chester Ave., Cleveland, Ohio.

6 Central District, Empire State Section, University Club, 431 E. Fayette St., Syracuse, N. Y. Senior night and Junior awards.

10 Metropolitan District, Empire State Section, Stouffer's, 100 E. 42nd St., New York, N. Y.

10 Lake States Section, Conway Hotel, Appleton, Wis., C. J. West Memorial Meeting. Tour of facilities of The Institute of Paper Chemistry, "What Should Research be Doing for the Pulp and Paper Industry?"

12 Ohio Section, Manchester Hotel, Middletown, Ohio. "Scientific Approaches to Papermaking," by Jasper Mardon, Oxford Paper Co., W. Carrollton, Ohio.

12 Northern District, Empire State Section, Hotel Woodruff, Watertown, N. Y. "Wet-Strength Resins for Paper."
Joint meeting with Beaver Falls Chapter A. I. C.

17 Indiana District, Ohio Section, Marott Hotel, Indianapolis, Ind.

19 Eastern District, Empire State Section, Queensbury Hotel, Glens Falls, N. Y.

27 Delaware Valley Section, Riegel Ridge Community House, Milford, N. J. Visit to mill of Riegel Paper Corp., Milford, N. J. 2 p.m.

May, 1962

1 Chicago Section, Chicago Bar Association, 29 So. La Salle St., Chicago, Ill. "Electro-types, Plate Making."
1 Golden Gate Section, San Francisco, Calif. R. M. True Memorial Lecture by K. Sarkanen, Finland.
4 Central District, Empire State Section, University Club, 431 E. Fayette St., Syracuse, N. Y. Dinner-dance.
5 Western District, Empire State Section. Dinner-dance.
10 Ohio Section, Hartwell Recreation Hall, Cincinnati, Ohio. Dinner-dance.
10 Northern District, Empire State Section, Woodruff Hotel, Watertown, N. Y. Dinner-dance.
11–12 Gulf Coast Section, Edgewater Gulf Hotel, Edgewater Park, Miss. Annual meeting.
15 Berkshire District, New England Section, Country Club of Pittsfield, Mass.
15 Lake States Section, Holiday Inn, Madison, Wis. "Forest Genetics." Visit to Forest Products Laboratory.
17 Pacific Section, Gearhart Hotel, Gearhart, Ore. Annual meeting. Joint meeting with Pacific PIMA.
18–19 Virginia-Carolina Section, Canton, N. C. Visit to mill of Champion Papers, Inc.
25 Metropolitan District, Empire State Section.
25 26 Southeastern Section, Wanderer Motel, Jekyl Island, Ga.
26 Lake Erie Section, Cleveland, Ohio. Annual meeting.

June, 1962

1–2 New England Section, Chatham Bar Arms, Chatham Bar, Mass.
7–9 Empire State Section, Whiteface Inn, Whiteface, N. Y. Maine-New Hampshire Section. (To be announced).

New England and Maine-New Hampshire

The Maine-New Hampshire and New England Sections of TAPPI held a joint spring meeting at Wentworth-by-the-Sea, Portsmouth, N. H., on June 10 and 11, 1961. Over 350 were present.

The New England Section elected the following officers:

Section Chairman: John W. Wendell, Jr., Tileston Hollingsworth Co.
Section Treasurer: Philip Slayton, Monsanto Chemical Co.
Section Secretary: Thomas C. Morganstern, Minerals & Chemicals Philipp Co.
Berkshire District Chairman. Milton J. Gordon, P. J. Schwietzer Co.
Berkshire District Vice-Chairman: William A. Hosmer, Hurlbut Paper Co.
Berkshire District Treasurer: Arthur W. Pinkham, Jr., Curtis-Wright Corp.
Berkshire District Secretary: Robert M. Henderson, E. D. Jones Corp.
Pioneer Valley District Chairman: Alexander E. Hutton, Jr., Fitchburg Paper Co.
Pioneer Valley District Vice-Chairman: David Habib, Technifax Corp.
Pioneer Valley Treasurer: William F. Nye, Jr., The Hubinger Co.
Pioneer Valley Secretary: Clifford S. Reppe, Hercules Powder Co.
Bay District Chairman: Albert C. Lamoreaux, Dennison Mfg. Co.
Bay District Vice-Chairman: David W. Lovering, Arthur D. Little, Inc.
Bay District Treasurer: William P. Stillman, Bird & Son.
Bay District Secretary: Paul Crane, Oxford Paper Co.

The Maine-New Hampshire Section elected the following officers:

Chairman: Whitney Osgood, S. D. Warren Co.
Vice-Chairman: Lawrence R. Adkins, Waterfalls Tissue Corp.
Secretary-Treasurer: Donald Bail, Great Northern Paper Co.
Executive Committee—three years:
H. E. Pratt, Pejepscot Paper Co.
John C. Cousins, Penobscot Chemical Fibre Co.
Clayton T. Backus, University of Maine.
Malcolm C. White, Oxford Paper Co.
Executive Committee—one year:
Edward N. Poor, Hudson Pulp & Paper Co.
Paul J. Shirley, Pennick & Ford Co.

The technical program Friday afternoon with John Wendell as moderator consisted of the following three papers:

1 "Peroxide Bleaching," by Robert Kindron, Ford Machinery and Chemical Corp.
2 "Hydrosulfite Bleaching," by Robert Barton, Virginia Smelting Co.
3 "Chlorine Dioxide Bleaching," by Andrew Garde, Versenes Inc.

At the technical session Saturday morning Edward N. Poor was moderator and the four papers presented were:

1 "Economic Justification of Instruments," by James Overall, Fischer & Porter Co.
2 "Continuous Freeness Recorder—Controller," George Harman, and Herbert Griffith, Bailey Meter Co.
3 "Application and Limitations of Beta Gage," by Robert Nero, Tracer Labs.
4 "Chemically Beaten Pulp," by Herbert Freedman, Penobscot Chemical Fibre Co.

Friday afternoon, the ladies enjoyed a series of book reviews by Mrs. Marion Rudken.

A reception at 6:30 p.m. was followed by the banquet and dancing in the evening. A midnight snack ended the official activities for the day.

In spite of heavy rain and fog Saturday a surprising number of scores were turned in for the golf tournament, and the tour of historical homes was well attended. The less hardy souls entered the bridge tournament or played bingo.

The Awards Dinner in the evening was followed by dancing until midnight.

Pulp Bleaching with Peroxide

R. R. Kindron and F. J. Rosebush

The role of peroxide in the bleaching of wood pulp is presented. It is shown that peroxide is one of the most versatile pulp bleaching agents available today, as it finds application in the bleaching of mechanical, chemi-mechanical as well as chemical pulps. Peroxide is not limited in its method of application but rather the method of application can usually be tailored to meet the mill's requirements.

Peroxide was originally introduced to the pulp industry as a bleaching agent for groundwood. Today, peroxide is still the prime agent for groundwood bleaching, however, because of its versatility, it finds wide application in the bleaching of chemical as well as chemi-mechanical pulps.

The versatility of hydrogen peroxide is exemplified by its accepted role as a bleaching agent for practically every type of pulp produced as well as by the wide variety of its methods of application.

Hydrogen peroxide is being used in the conventional tower bleaching method, at moderate or high density, as well as in the more specialized areas as the cold steep, drier bleaching and refiner bleaching applications.

The method and type of peroxide application is, of course, dependent on pulp type, desired brightness, and equipment or capital available for the bleaching operation.

R. R. Kindron and F. J. Rosebush, Becco Chemical Div., Food Machinery and Chemical Corp., Princeton, N. J.

Table I. Groundwood Bleaching Systems

Wood species	Bleaching consistency, % O.D.	H_2O_2 60%, 1%	Temp., °F.	Brightness, G.E. Unbleached	Brightness, G.E. Bleached	Second stage Hydro, %	Second stage Brightness
90% Spruce-balsam, 10% Poplar	24	0.47	116	61.1	71.7	...	...
	22	0.94	115	61.1	77.0	1.0	78.3
	15	0.50	121	61.1	69.8	...	...
	15	1.00	118	61.1	73.0	...	...
100% Spruce-balsam	13	1.10	100	61.8	74.0	0.9	78.0
100% Southern pine	12	1.00	120	55.6	67.9	1.0	71.1
	12	1.00	110	62.0	69.5	...	...
	25	1.00	110	62.0	71.9	...	...
Western hemlock	14	1.50	140	54.8	68.4	1.0	72.0
	14	1.20	140	54.8	66.1	1.0	69.4

GROUNDWOOD

Normally, bleached groundwood brightnesses in the mid 60 to low 70 level are required for the production of certain groundwood specialty papers. Usually, these brightnesses are achieved with peroxide in a moderate density (10 to 14%) tower bleaching application. In the past few years, there has been an increasing demand for groundwood brightnesses approaching and exceeding the 75 G.E. level.

To achieve these higher brightness levels, it becomes evident that certain modifications are necessary to improve the efficiency of the bleaching operation. These improvements or modifications logically start with the raw material, namely, the wood. It has been known for years that improved bleach response could be obtained when fresh, sound wood was used. It has not been until recently that the desire to maintain reduced wood yard inventories had made it possible to obtain a relatively continuous supply of fresh, sound wood for grinding. The improved control in the wood yard has resulted in increasing the unbleached groundwood brightness from the 55 to 57 G.E. level to the 60 to 62 level with a resultant increase in bleached brightness.

Various pulp pretreating agents have been used successfully in the past to help improve the bleaching efficiency when old, dark, or pithy wood was used. Usually, no advantage was noted from the use of these agents when fresh wood was used.

Today, more selective agents such as tetrasodium salt of ethylenediaminetetraacetic acid (EDTA), pentasodium salt of diethylenetriaminepentaaectic acid (DTPA), etc., are coming into wider use to help remove or tie up the heavy metal contaminants, such as copper, manganese and iron, found in the groundwood system. The removal of these metals assists in increasing the efficiency of the groundwood bleaching operation by decreasing the peroxide lost to decomposition.

The improvement in wood quality and the use of selective pretreating agents usually improves the bleach response in a moderate density peroxide stage by two to three brightness units. However, to achieve the very high brightness demanded in some operations, additional modifications have been proposed. One of these is the addition of a hydrosulfite stage after the neutralization step following a moderate density peroxide stage. Usually, this stage is conducted around

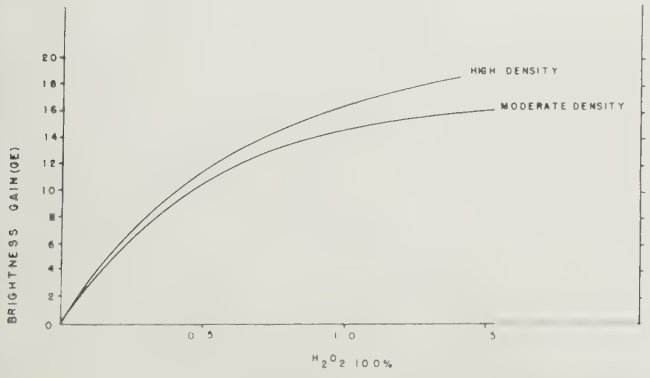

Fig. 1. Comparison moderate density versus high density

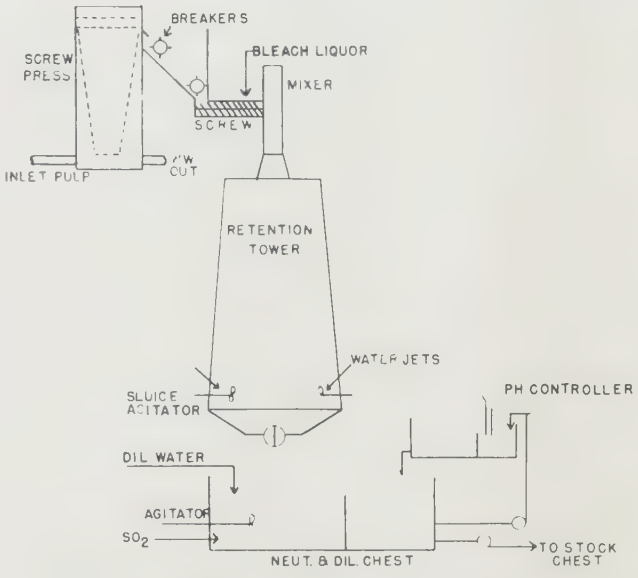

Fig. 2. Continuous high density tower system

140°F. at low or moderate density using approximately 1% hydrosulfite with or without the addition of sodium tripolyphosphate or tetrasodium salt of ethylenediaminetetraacetic acid (*1, 2*).

The brightness gain from the second stage will depend on the brightness obtained from the first-stage peroxide application. Approximately 5 additional brightness units will be obtained on 70 G.E. peroxide bleached groundwood with the gain from the second stage diminishing as the brightness from the first stage increases.

Another modification to the bleaching process proposes increasing the density of the peroxide stage to 20% or greater. In the single stage high density peroxide application, because of the reduced liquor to fiber ratio, the efficiency of the system increases, thus making it possible to bleach the groundwood to higher brightness levels at greater chemical economy than is usually possible by other methods.

Recent improvements in the design of various pulp dewatering presses have materially aided in making the peroxide high density crumb bleaching method commercially feasible.

Examples of the various groundwood bleaching systems are presented in Table I. Figure 1 compares moderate and high density peroxide bleaching methods. A typical single stage high density bleaching system is presented in Fig. 2. Minor modifications of this system are shown in Fig. 3; elimination of the bleaching tower and addition of an air system to transfer the impregnated high density crumb stock to boxcars, trucks or bins, adapts the system to the high density cold steep bleaching process wherein bleaching is accomplished while the pulp is in transit or in storage at ambient temperature (*3*). In the past year, two new high density peroxide bleaching systems have gone into commercial operation on groundwood. One system utilizes the high density tower application, the other the crumb cold steep bleaching process.

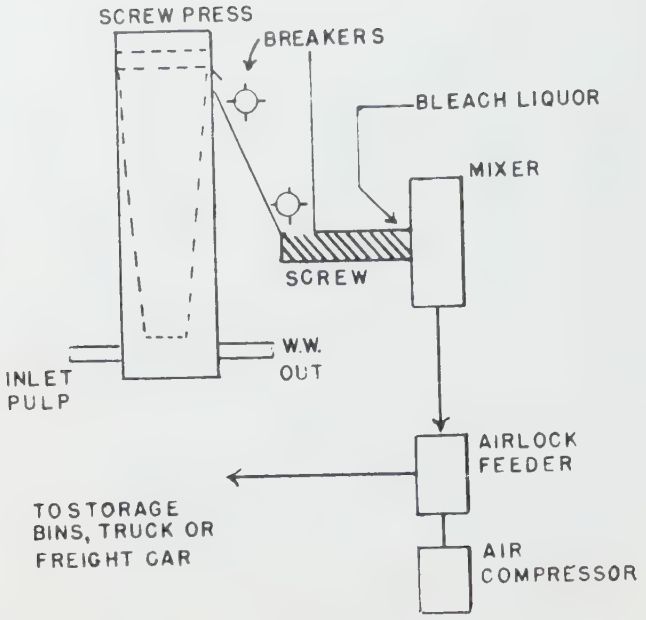

Fig. 3. Steep bleaching of pulp crumbs

Ultimately, the method adopted by a mill for bleaching groundwood, or in fact any pulp, will be dependent on the factors cited previously, namely, wood species, desired brightness levels as well as equipment and/or capital available for the bleaching process.

CHEMICAL PULPS

The inclusion of a peroxide stage in several commercial chemical pulp bleaching installations has helped satisfy the increasing demand for high brightness pulps possessing good brightness permanence and maximum strength characteristics. The peroxide application is usually made in the final stage of a bleaching sequence, however, there are instances where it is used to advantage in an intermediate stage.

Aside from its use in the bleaching tower, hydrogen peroxide finds wide use in the pulp drier steep bleaching application (*4*) by mills producing market pulp. This system overcomes the brightness loss usually experienced in drying and baled storage and does so with a minimum of additional capital expenditures for equipment. Bleaching chemicals, usually 0.05 to 0.25% H_2O_2 100% and a soluble phosphate, are applied to the pulp web prior to its entry into the drier section. It becomes apparent that such an application offers considerable flexibility of operation in that it can be used for special orders without altering operation of the bleach plant and the subsequent production of transition pulp. Typical results of the peroxide drier steep bleaching application are presented in Table II.

Table II. Multistage Kraft Bleaching—Drier Steep Bleaching

Wood species	*Bleaching sequence*[a]	*H_2O_2 100%, %*	*Brightness, G.E.* After drier	Bale aged, days		*Viscosity*[b] *0.5M Cu(En)₂*
Southern pine	C-E-H	...	66.2	66.6	7	13.8
	C-E-H-P	0.19	72.6	74.1	7	12.6
Southern pine	C-E-H	...	74.7	73.8	35	13.3
	C-E-H-P	0.26	78.3	83.6	35	11.3
Southern hardwood	C-E-H-H	...	83.7	81.7	35	6.8
	C-E-H-H-P	0.17	84.5	85.6	35	6.0
Southern hardwood	C-E-H-D-D	...	82.4	81.3	14	...
	C-E-H-D-D-P	0.19	85.4	88.4	14	...
	C-E-H-D-D	...	87.0	88.4	9	...
	C-E-H-D-D-P	0.12	89.7	91.4	9	...
Southern pine	C-E-H-D	...	85.0	82.7	42	10.7
	C-E-H-D-P	0.08	87.1	86.2	42	8.8
	C-E-H-D	...	87.1	79.3	42	12.4
	C-E-H-D-P	0.15	87.3	86.8	42	12.0

[a] C, Chlorination. E, Caustic Extraction. EH, High pH Hypochlorite. H, Hypochlorite. D, Chlorine Dioxide. P, Peroxide.
[b] Viscosity determinations made on aged samples.

Kraft

In recent years, there have probably been more investigations on the bleaching of kraft pulp than any other type. Because of the large capital expenditure necessary for multiple bleaching stages, the search has been for systems capable of producing high brightness pulp with the least number of stages. The advent of chlorine dioxide made it possible to reduce the number of stages, however, it was later found that the inclusion of a peroxide stage in the bleaching sequence enhanced the properties of chlorine dioxide bleached pulps (*5, 6*).

Continued efforts by numerous workers to reduce the number of bleaching stages has brought forth an interesting modification which combines the caustic extraction and hypochlorite stages (*7*) in the kraft bleaching sequence. Expansion of this work in our laboratories has shown that addition of a peroxide stage to this sequence makes it possible to bleach a kraft pulp in four stages (C-(EH)-D-P) to brightnesses in excess of 85 G.E. maintaining good strength characteristics and improving the brightness stability of the bleached pulp.

The results of our laboratory study evaluating various bleaching sequences, including the modified four-stage sequence, are presented in Table III. The results of the four stage sequence compare very favorably with those obtained in five or more bleaching stages.

Sulfite

Usually sulfite pulps are bleached to relatively high brightness levels in two or more bleaching stages. As with groundwood and kraft pulps, peroxide can be applied to advantage in a variety of ways to improve pulp brightness and brightness stability. Peroxide finds application in single stage bleaching where moderate brightnesses and high bleached yields are desired. Where brightnesses in the mid 80 G.E. range are desired, it can be used as a second stage following a

Table III. Multistage Kraft Bleaching—Moderate Density Tower

Unbleached pulp	*Bleaching sequence*[a]	*Cl_2, %*	*NaOH, %*	*Av. Cl_2, %, as hypo*	*ClO_2, %*	*H_2O_2 100%, %*	*Brightness*	*Heat aged 105°C., 1 hr.*	*Viscosity, 0.5M Cu(En)₂*
Northern pines	C-EH-D	5.0	2.0	2.5	1.0	...	79.4	72.0	21.0
K no. 16.9	C-EH-D-P	5.0	2.0	2.5	1.0	0.25	86.5	83.6	18.7
Visc. 27.1	C-EH-D-P	5.0	2.0	2.5	1.0	0.50	88.8	86.4	18.6
Jack pine	C-E-H-D	7.0	2.0	2.0	1.0	...	81.6	73.9	12.2
K no. 17.4	C-E-H-D-P	7.0	2.0	2.0	1.0	0.22	85.5	82.1	10.4
Visc. 24.9	C-EH-D	7.0	2.0	2.0	1.0	...	76.7	70.1	19.3
	C-EH-D-P	7.0	2.0	2.0	1.0	0.22	84.3	81.2	17.6
	C-EH-D-P-H	7.0	2.0	2.3[b]	1.0	0.22	89.4	85.4	14.6
Northern hardwoods									
K no. 10.5	C-E-H-P	4.0	2.0	2.0	...	0.25	85.6	84.2	7.1
Visc. 20.5	C-E-D-P	4.0	2.0	...	0.75	0.38	89.0	86.7	15.2

[a] C, Chlorination. E, Caustic extraction. EH, High pH hypochlorite. H, Hypochlorite. D, Chlorine dioxide. P, Peroxide.
[b] 2% Av. Cl_2 in EH stage, 0.3% av. Cl_2 in final hypo stage.

Table IV. Sulfite Bleaching with Peroxide

Bleaching sequence[a]	H_2O_2 100% %,	Brightness, G.E. Unbleached	Before H_2O_2	After H_2O_2	Aged		Viscosity, 0.5M Cu(En)₂
			Cold steep				
P	0.50	59.0	59.0	75.7	76.0	1 mo. bale	.
P	0.70	59.9	59.9	79.4	81.4	1 mo. bale	...
			Drier Bleach				
C-E-H-D	...	60.1	86.6	...	84.4	1 mo. bale	...
C-E-H-D-P	0.08	60.1	86.6	88.0	88.4	1 mo. bale	...
			Moderate Density Tower				
C-E-H	...	59.8	84.8	...	80.2	1 hr. at 105°C.	15.7
C-E-H-P	0.125	59.8	84.8	88.2	87.3	1 hr. at 105°C.	15.4
C-E-H	...	60.8	85.9	...	80.8	1 hr. at 105°C.	12.8
C-EH-P	0.125	60.8	83.9	87.2	84.6	1 hr. at 105°C.	19.4
C-E-H	...	58.5	87.2	...	70.8	1 hr. at 105°C.	11.2
H-P	0.50	58.5	...	87.3	79.5	1 hr. at 105°C.	19.2
P	0.825	53.8	53.8	71.3	...	...	...
P	1.00	53.8	53.8	75.4	...	...	...

[a] C, Chlorination. E, Caustic extraction. EH, High pH hypochlorite. H, Hypochlorite. D, Chlorine dioxide. P. Peroxide.

Table V. Multistage Bleaching of Neutral Sulfite Semichemical Pulp

Unbleached pulp	Bleaching sequence[a]	Cl_2, %	NaOH, %	Av. Cl_2,%, as hypo	H_2O_2 100% %,	Brightness, G.E. Bleached	Heat aged 105°C., 1 hr.	Viscosity, 0.5M Cu(En)₂
Brightness 49.2	H-P	...	...	19.0	0.37	89.2	85.5	8.2
Visc. 8.2	C-E-P	17.5	4.0	...	1.0	87.6	83.1	10.0
	C-EH-P	17.0	1.0	1.5	0.75	87.3	82.6	10.2
	C-H-P	16.0	...	2.0	0.83	88.2	85.2	10.4
Brightness 44.1	H-P	...	...	11.0	0.38	82.2	78.4	12.6
Visc. 5.6	C-EH-P	10.0	1.5	4.0	0.25	82.8	78.4	12.1
	C-E-H	10.5	2.0	4.5	...	83.7	74.9	9.6
	C-P-H	9.5	...	3.0	0.25	84.0	77.1	10.8
	C-E-H	11.5	3.0	1.0	...	88.4	81.0	9.7
	C-E-H-P	11.5	3.0	1.0	0.125	91.5	89.4	7.8

[a] C. Chlorination. E, Caustic Extraction. EH, High pH Hypochlorite. H, Hypochlorite. D, Chlorine Dioxide. P, Peroxide.

first stage hypochlorite. For brightnesses in excess of 85 G.E., peroxide can be applied in the final stage of a multistage sequence.

Presented in Table IV are results of these various peroxide applications.

NEUTRAL SULFITE

Peroxide is advantageous also in the bleaching of neutral sulfite semichemical pulps. With the neutral sulfite pulps, it is usually beneficial to precede the peroxide stage with one or more bleaching stages to help satisfy the high chemical demand of these high yield pulps. As is shown in Table V, peroxide bleaching materially assists in improving the brightness and brightness stability with minimum effect on resulting strength properties of the neutral sulfite pulps.

COLD SODA

Akin to neutral sulfite is cold soda pulp. Early bleaching studies showed that chlorine compounds turned most cold soda pulps a distinct yellow color which was particularly difficult or impossible to overcome in subsequent bleaching stages. It was found that peroxide or a combination peroxide-hydrosulfite treatment improved the pulp brightness without yellowing.

Table VI. Cold Soda—Refiner Bleaching

Wood species	H_2O_2 100% %,	Refiner temp., °F.	Brightness, G.E. Unbleached	Retention time 15 min.	Retention time 30 min.
Aspen					
Old wood 1 year	2.1	200	31.0	50.5	50.0
Fresh peeled, less than 3 months	2.1	200	37.0	64.3	71.8
Fresh peeled, less than 3 months	1.3	200	37.0	61.0	66.7
Fresh peeled, less than 3 months	0.8	200	37.0	56.0	62.0
				After refiner	Two hours
Northern hardwoods					
Birch, beech, maple	1.0	180	39.7	54.1	62.9
	1.5	180	39.7	56.3	64.0

As a result of intensive investigation, the Becco Chemical Division developed a method for bleaching cold soda in the chip refiner. This bleaching method, commonly referred to as refiner bleaching (*8*), takes advantage of the mixing action of the refiner as well as the heat developed during refining. A short retention period, as in a stock chest, after refining will usually suffice to develop maximum brightness from this type of application.

The refiner bleaching technique makes it possible for a mill

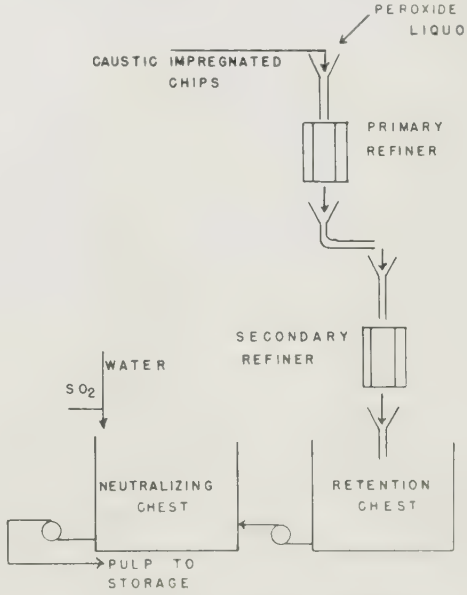

Fig. 4. Refiner bleaching

Table VII. Cold Soda—Tower Bleaching

Wood species	Pretreatment	Bleaching consistency, % O.D.	Brightness, G.E. Unbleached	Brightness, G.E. Bleached	2nd stage 1% hydro, 3.0% consistency. 140°F., 1 hr.
A[a]—Northern oaks	None	12	34.7	62.0	65.8
	SO_2pH 2.0	12	34.7	64.0	68.8
	None	25	34.7	64.0	67.7
	SO_2pH 2.0	25	34.7	71.8	73.3
B—Southern hardwoods: oak, gum, poplar, magnolia	None	25	42.4	62.8	69.1
	H_2SO_4pH 3.0	25	42.4	69.5	73.0
	H_2SO_4pH 3.0 + 0.1% EDTA	25	42.4	70.1	74.5
C—Northern hardwoods: birch, beech, maple	None	12	40.7	58.8	63.7
	Wash	12	40.7	63.8	68.6
	Wash + SO_2pH 2.5	12	40.7	71.4	73.2
	Wash + 0.1% EDTA	12	40.7	69.5	72.7
	None	25	40.7	64.4	72.2
	Wash	25	40.7	72.7	74.9
	Wash + SO_2pH 2.5	25	40.7	78.1	79.2
	Wash + 0.1% EDTA	25	40.7	76.2	79.2
D—Cottonwood	None	10	43.0	63.5	...
	0.2% DTPA H_2SO_4pH 4.0	10	43.0	67.7	70.8
		10	43.0	66.6	68.6

[a] A—1.75% H_2O_2 100%, 160°F., 3 hr.; B—1.50% H_2O_2 100%, 160°F., 3 hr.; C—1.50% H_2O_2 100%, 160°F., 3 hr.; D—1.50% H_2O_2 100%, 180°F., 3 hr.

to bleach most cold soda pulps to the 60 to 65 G.E. brightness level with a minimum capital expenditure for additional equipment as this method obviates the need for chemical mixers, a bleaching tower, and considerable additional steam. A schematic flow plan for the refiner bleaching method is shown in Fig. 4. Results of the refiner bleaching application are presented in Table VI.

For mills desiring cold soda pulp with brightnesses in excess of 65 G.E., the bleaching operation becomes somewhat more involved in that mixers, bleaching towers, pulp thickeners, etc., are required. Our investigations have shown that it is possible to bleach beech, birch and maple hardwood cold soda pulps to brightnesses approaching the 80 G.E. level in either a single-stage high density or a two-stage moderate density peroxide-hydrosulfite application. Usually a pretreatment of the washed unbleached stock with either acids, reducing agents or chelating agents, prior to the peroxide application, is advantageous in obtaining maximum brightness response from the bleaching operation. The results of various bleaching studies conducted in our laboratories are presented in Table VII. Again, it will be noted that the response obtained from a second stage hydrosulfite application decreases as the brightness response from the first stage peroxide application increases.

Independent of the bleaching procedure adopted, in order to obtain maximum brightness response, it is of utmost importance that fresh wood be used for the production of cold soda pulp.

CONCLUSIONS

It has been shown that hydrogen peroxide can be used effectively in a single stage or in a multistage bleaching sequence and further that hydrogen peroxide finds application in the bleaching of mechanical, chemi-mechanical, and chemical pulps. It can truly be said that hydrogen peroxide is one of the most versatile bleaching agents available today.

LITERATURE CITED

1. Sparrow, D. B., U. S. pat. 2,707,144 (1955).
2. Sparrow, D. B., Moylan, J. E., and Britt, K. N., U. S. pat. 2,707,145 (1955).
3. McEwen, R. L., U. S. pat. 2,513,344 (1950).
4. Sheldon, F. R., McEwen, R. L., and Price, C. E., U. S. pat. 2,822,236 (1958).
5. Casciani, F. and Storin, G. K., *Tappi* 33: 588 (1950).
6. Casciani, F. and Storin, G. K., U. S. pat. 2,494,542 (1950).
7. Rapson, W. H. and Anderson, C. B., *Tappi* 41: 486 (1958).
8. Jahne, J. F. and Price, C. E., *Tappi* 43: 226A (1960).

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Chelating Agents in Papermaking

Andrew J. Gard

Chelation is analogous to the titration of an acid by a base; the metal ion is considered an acid and the chelating agent a base. The chemistry of the reaction is given. Its application to pitch control is mentioned and some data are given on the use of a chelating agent in bleaching in the prevention of color reversion.

A FEW papers on pulp bleaching have made reference to EDTA and the deactivation of metal ions. The reasons for its use are well recognized but the mode and complexity of the task performed by EDTA and analogous compounds has not been offered to the technical personnel of the paper making industry. We wish to review some of the aspects of chelation chemistry to papermaking.

CHEMISTRY

Chelation is analogous to the titration of an acid by a base; the metal ion is considered an acid and the chelating agent the base. Chelating agents act as donors of electrons and metal ions as acceptors; so the process is one of neutralization. Generally speaking, the higher the valence of the metal ion the greater the tendency for chelation to take place. Thus, trivalent ions, such as ferric ion, would be chelated before divalent ions, such as copper, manganese, or calcium. The order of chelation of metal ions is trivalent, then divalent heavy metal ions, and finally alkaline earth ions.

Aluminum ion does not lend itself to chelation by EDTA or its derivatives because it hydrates with water molecules quite strongly and the pH of the system must be less than 3.5 before a complex with Al_3^{+++} ion is formed. For this reason, preferential chelation of iron in alum solutions is possible and we can supply information on the comparative cost of converting commercial alum to the iron free grade. The fact that EDTA cannot tie up aluminum ion eliminates any interference with sizing as occurs with the polyphosphates.

To explain the stability of the metal chelates and the deactivation effect by EDTA, we must examine the structure of the chelating agent molecule, which is a symmetrical arrangement of hydrogen, carbon, nitrogen, and oxygen atoms.

Figure 1 illustrates the chelation of cupric ion by the simplest protein molecule, glycine showing the formation of a five membered ring structure with the metal ion. The illus-

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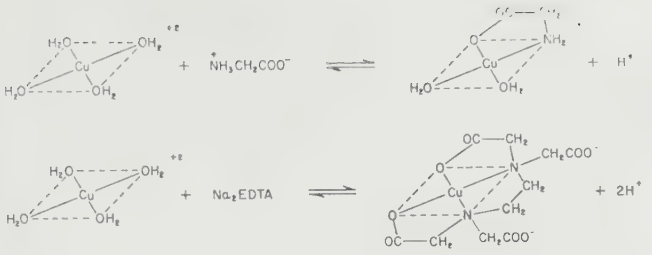
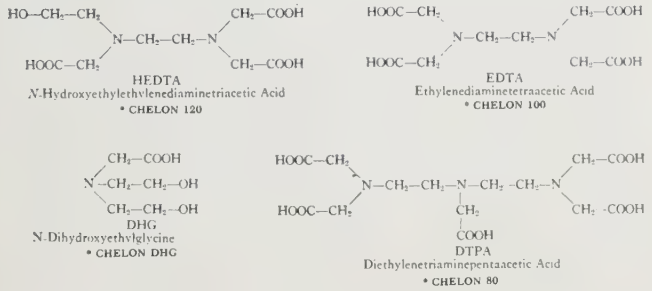


Fig. 1

tration showing EDTA chelation of cupric ion shows the formation of four five-membered ring structures and that multiring formation conveys extraordinary stability to the metal chelate. This is basically why EDTA chelation of metal ions finds such a diverse use in medicine, agriculture, chemical, and industrial cleaning industries. By definition, a chelate structure is characterized by formation of more than one bond between the metal ion and the molecule of a complexing agent with the formation of an inner-ring structure incorporating the metal ion as an integral part of this ring structure. The metal chelate resulting from this combination is a completely new entity of exceptional stability.

All metal chelates of EDTA or its derivatives become less stable at low pH values; that is, less than 3.0, and when the alkalinity of the aqueous system is off the pH scale. This is true when the mole to mole ratio of metal ion to chelating agents is considered, if an excess of the chelating agent is present, more severe alkaline conditions are required to hydrolyze the metal chelate. In the case of iron, EDTA cannot effectively bind iron above a pH of 7.5 unless progressively high excess concentrations are employed. Other chelating agents have been devised for this specific purpose and they are HEDTA, DTPA, and DHEG.

Figure 2 shows the structure of each of the current com-



* Trade Mark of the Chelate Chemical Corp.

Fig. 2

mercially available chelating agents and the Chelon labels for each. Table I compares the Log K values (stability constants) for the commercially available chelating agents showing DTPA to be superior. These values give some idea of the strength of the metal chelate but do not predicate the behavior in a competitive aqueous media.

Table II shows the order of increasing efficiency of these chelating agents toward iron and calcium with pH as the variable. The chelating agents other than EDTA have a hydroxyl grouping or an additional functional carboxyl group to preclude hydrolysis of ferric ion to ferric hydroxide in the alkaline range. Prevention is one thing and redissolving iron

Table I. Stability Constants*

Ligand	*Log $KFe^{+3}A$*	*Log KcuA*	*Log KcaA*
DHG	?	8.15	...
HEDTA	19.6	17.4	8.0
EDTA	25.1	18.8	10.6
DTPA	28.6	21.03	11.0

* Organic Sequestering Agents, Chaberek and Martell, John Wiley & Sons, Inc., N. Y., N. Y.

Table II. Effect of pH on Fe^{+3} Chelation

pH	*Order of Increasing efficiency*
2–3	HEDTA < DTPA < EDTA
4.0–8.5	HEDTA < EDTA < DTPA
8.5–10.0	EDTA < HEDTA < DTPA

Effect of pH on Calcium Chelation

Ligand	*pH Range of 90–100% chelation*
HEDTA	>8.0
EDTA	>7.0
DTPA	>6.5

hydroxide is another. Chelating agents are more effective in prevention than in dissolution of precipitated iron. Another fact is the removal of ferric ion from cellulose; which binds ferric ion very strongly, requires either an excess of the chelating agent, time of contact or in combination with a reducing agent such as hydrosulfite to strip the fixed iron from cellulose. Another interesting fact is that high calcium ion concentration can displace a heavy metal ion from its chelate structure with EDTA but this is not the case with DTPA or HEDTA. There have been many laboratory evaluations made with EDTA that have resulted in negative results for lack of this information. This is one reason why DTPA, our Chelon 80, the newest of the family, has played such a prominent part in peroxide pulp bleaching.

The piecemeal offerings in technical publications on applications of EDTA in papermaking distort its true significance. Pulp mills are constantly making refinements in plant equipment to preclude metallic contamination; but iron, copper, manganese, zinc, and calcium are associated with the tree's metabolism plus the pick-up due to corrosion, water supply, and chemical additives, make up heavy metal ion concentrations that create problems with pitch ingredients, bleaching chemicals and brightness stability of the finished product.

APPLICATIONS

Swanson, in the January issue of *Tappi*, suggests EDTA as an additive to the shower water at the grinders. This is one application we have pioneered since 1952, and the data of Table III shows what can be expected from 2.5 to 5 lb. of Chelon 100 or Chelon 80 per ton of o.d. pulp introduced directly into the shower water or on the stone surface. Swanson's suggestion pertained to pitch control but EDTA or DTPA will keep the stone surface clean; call it chemical etching if you wish, but with pocket grinders we have shown an increase in brightness, more so with old wood than green wood. With continuous grinders such as the Roberts' grinder, we have conducted extensive tests resulting in about 2 points increase in brightness both on spruce and jack pine.

Table III. Groundwood Additives—Great Northern Grinder

First Trial	*Untreated*	*5 lb./ton Na_4EDTA (38% sol.)*	*10 lb./ton Na_4EDTA (38% sol.)*
pH	4.88	4.88	4.95
Consistency, %	4.94	7.39	8.19
Freeness C.S., ml.	125	178	235
Brightness (screened)	53.7	59.7	59.3

Second Trial	*Brightness—G. E. Units Initial*	*Final*	*Gain*
Control "dirty wood"	51.4	...	...
+Na_4EDTA (38% Solution) at 3 lb./ton	...	57.6	6.2

Third Trial	*Temp., °F.*	*Consistency, %*	*Freeness*	*Brightness G. E. units*
Control	160	6.72	84.2	54.0
+Na_4EDTA (38% solution) at 3 lb./ton	158	4.93	83.8	57.5

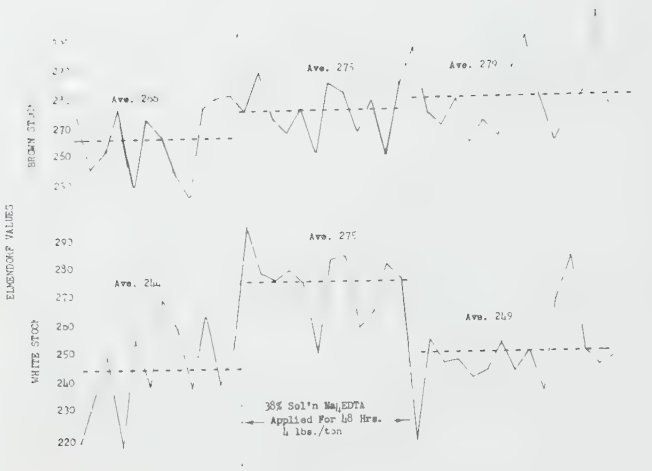


Fig. 3

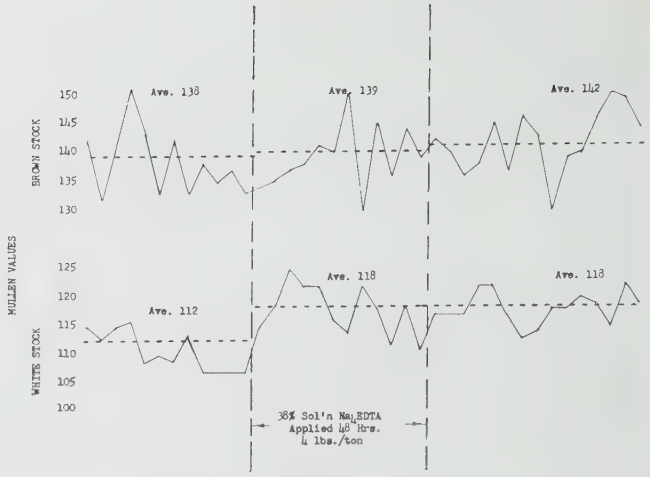


Fig. 4

However, temperature plays a big part in this action of EDTA or DTPA on brightness and grinding efficiency.

Pitch control with EDTA for mechanical or chemical pulps was first described by Gustafsson (*1*) when he reported on the effect of EDTA in reducing the adherence of pitch to copper metal surfaces. His screening technique illustrates the fact that EDTA is adsorbed on the copper metal surface and precludes pitch occlusion. When a change in pH of the pulp slurry from the alkaline to the neutral or acid range occurs in the presence of high calcium ion and proper temperature conditions, pitch particles become tacky and agglomerate. One could not afford to add EDTA on a mole to mole basis to tie up all the calcium ion content to prevent such agglomeration, but EDTA has been shown to be effective at 3 to 5 lb. per ton o.d. pulp as Chelon 100. Of course, it may not be the complete answer to pitch problems, but in conjunction with dispersing agents, more effective pitch control will be evident. Lignin sulfonates or the phenol-formaldehyde disulfonates are deflocculating agents that, when used in excess of 3 to 3 lb. per ton, have an adverse effect on size and filler retention. The use of these additives at 1 to 2 lb. per ton, plus a chelating agent, EDTA or DTPA, should result in more effective control of pitch agglomeration without aggravating retention of size and filler through the machine. Glassine mills use such a combination in their beater furnish and to the fresh water at the machine chest. For chemical pulps, application at the brown stock washer or at the caustic extraction stage will help wash out the pitch and also reduce the tendency to agglomerate.

Another area of application for chelating agents is in peroxide and hydrosulfite bleaching of groundwood. DTPA, Chelon 80, proved to be more effective than either tripolyphosphate or EDTA in stabilizing the pulp slurry to the peroxide bleach liquor. The real value of DTPA in peroxide bleaching has been in the improved brightness stability of the bleached groundwood. Mills that are using tripolyphosphate or had discontinued its use found when they tried Chelon 80 that peroxide residuals at the bottom of the tower increased 5 to 8 fold above normal. In some mills an increase in brightness of 2 to 3 points occurred but the significant feature was the improved stability of the bleached stock. Very little color reversion occurred in storage or through the machine. One mill reduced the bleached sulfite in the beater furnish from 20% to 5% and the "off-machine" brightness remained within specifications. Another mill reduced the amount of peroxide by 1/4% and produced the same brightness "spring" with healthier peroxide residuals and greatly improved color stability even with 5-hr. retention time. These are some of the manifestations of Chelon 80 in peroxide bleaching of groundwood. The improved color stabilizing action also applies to hydrosulfite bleaching of groundwood.

A recent development is the improved stability or color yield resulting in the use of EDTA or DTPA with optical brighteners. Less optical dye is required when the chelating agent is added to the pulp slurry ahead of the point of dye application.

A number of chemical pulp mills employ DTPA to prevent color reversion of their bleached stock. Single stage chlorine dioxide bleached pulps suffer from poor color stability and various means are used to improve the final brightness. One method involves the use of peroxide plus a polyphosphate and DTPA sprayed onto the wet web of the sheet ahead of the drier. Another mill adds the DTPA to the SO_2 antichlor stage followed by a brief wash cycle to remove the ferric ion and thus effect better color stability. Removal or reduction of ferric ion content of the bleached pulp improves color stability.

One misleading feature of laboratory color stability tests is the use of a UV fadeometer. The iron chelate of EDTA or DTPA is photochemically reduced or degraded by extended exposure but this is usually not the case on plant pulp production where an excess of calcium ion is prevalent. The iron chelate of DTPA is stable in the presence of calcium ion and this same metal ion conveys UV stability to the iron-DTPA chelate. This is not true of EDTA.

We wish to introduce another area where chelation should prove its worth, and that is in hypochlorite bleaching. Back in 1940, Rue reported on metallic catalysts in hypochlorite bleaching but little attention has been given to this phase of chemical pulp bleaching. At this year's New York TAPPI meeting, Herbst reported on the catalytic effects of copper

Table IV. Spectrographic Analysis of Kraft Pulp

Metal ion, %	*Brown stock,*[a] %	*1st Stage acid Cl_2,* %	*2nd stage $Cl_2 + Ca(OH)_2$,* %	*3rd stage 1st hypo,* %	*4th stage NaOH,* %	*5th stage 2nd hypo,* %	*Antichloride,* %
Fe	0.0030	0.0009	0.0036	0.0040	0.0084	0.0046	0.0038
Cu	0.0003	0.0002	0.0002	0.0002	0.0048	0.0002	0.0002
Mn	0.0030	0.0002	0.0004	0.0005	0.0014	0.0005	0.0003
Ni	0.0006	0.0006	0.0004	0.0048	0.0096	0.0010	0.0003
Ca	0.15	0.15	0.87	1.4	1.0	0.92	0.44
Mg	0.13	0.020	0.034	0.055	0.048	0.054	0.020

The above analysis is on the oven-dry samples, sampled at the washer of each stage.
[a] Key 0.01% = 100 p.p.m.

and nickel in hypochlorite bleach liquors. These metal ions increased the bleach consumption and the rate of depolymerization or degrading of the pulp and were a distinct disadvantage in bleaching. We pursued this approach in 1956 and Table IV with Figs. 3 and 4 show the stabilizing action of EDTA.

However, the stabilizing action of EDTA was evident by the fact that there was no loss in Elmendorf tearing strength, whereas, before the trial started the loss ranged at 22 points from 266 to 244. The loss after the trial ended was from 279 on the unbleached to 249 on the bleached pulp, or 30 points. The burst values remained unchanged but viscosity values showed 9.6 before and 10.3 with EDTA reverting to 9.5 after the trial period.

These data warrant further exploration. The plaguing problem of brightness stability or color reversion of hypochlorite or single stage chlorine dioxide bleached pulps is generally attributed to the formation of oxycellulose. It is common knowledge that overbleaching results in formation of the carbonyl type oxycellulose which possesses a special tendency to yellow. The special tendency to yellowing is also associated with the hemicellulose content of the pulp; those high in hemicellulose have a greater tendency for color reversion than ones with a lower content. It must be kept in mind, however, that as the hemicellulose and lignin content are reduced the surfaces of cellulose may be oxidized to some extent to products causing color reversion.

Referring to Table IV, the metal ion contamination at the 4th stage caustic extraction shows the pulp contained 84 p.p.m. Fe, 48 p.p.m. Cu, 14 p.p.m. Mn, 96 p.p.m. Ni and 10,000 p.p.m. Ca. An overseer of bleaching in the textile industry would never attempt to bleach cotton greige goods with metal ion contamination of the magnitude shown at the fourth stage. It not only would result in a "poor bottom"—yellowish white—but the strength of the piece goods would be so greatly altered that the finished fabric would go into "seconds." Yet, the paper industry bleaches cellulose under such conditions.

These catalytic metal ions simply intensify the bleaching action and result in more extensive degradation than would occur without their presence. Their presence leads to surface oxidation of cellulose and products, oxycellulose, that produce color reversion. Of course, peroxide and borohydride are being offered to further modify these degradation products from the hypo stage to lessen the tendency to turn yellow, but this is post correction and at present fairly expensive. This problem of color stability warrants the use of chelating agents as a pretreatment to the hypo or chlorine dioxide stage. It should increase the washing efficiency of the chemical pulps or, stated another way, demineralize cellulose before the bleach stage.

It appears that DTPA, Chelon 80, is best suited for this application. We checked the stability of the commercially available chelating agents, namely, EDTA, HEDTA, DTPA and NTA (nitrilotriacetic acid) to a 0.1% sodium hypochlorite bleach liquor and also their copper and nickel chelates. 1×10^{-3} molar solutions of these chelating agents were used as well as the metal chelates and at a temperature of 90°C. only DTPA exhibited stability to hypochlorite. DTPA showed over 75% available calcium chelation after 3 hr. exposure. As the copper and nickel chelates, NTA is the least stable, showing complete destruction at 65°C. in less than ten minutes followed by HEDTA and EDTA. Again, DTPA held the copper and nickel in solution for over $^1/_2$ hr.

We are pursuing this stability project in a more extensive manner and should have adequate data on the efficiency of chelation and stabilization of the bleach liquor in the near future. However, the one plant trial conducted with EDTA and the results obtained suggest DTPA because of its greater stability to hypochlorite and the stability of the heavy metal chelates in the presence of high calcium ion. Actually, it is only the first 10 to 15 min. of the bleach cycle that available chlorine is at a high concentration when it is detrimental to the pulp and the chelating agent. We have shown that the chelating agent DTPA appears to withstand such severe conditions. We feel DTPA, Chelon 80, will be of benefit to all mills using a hypochlorite stage and a last stage chlorine dioxide bleach.

We have reviewed some of the chemistry of EDTA and its derivatives and the areas of application in pulp and paper production. EDTA and DTPA appear to be the more useful of commercially available chelating agents in applications discussed. We hope the technical and production personnel of the pulp and paper industry will be less reluctant to give chelators of metal ions, Chelons, an opportunity to perform in problems of making and marketing paper and paper products. Chelating agents may appear complicated but the purpose they serve is really a simple one.

LITERATURE CITED

1. Gustafsson, C., Tammela, V., and Lindh, T., Paper And Timber (Finland) 36, No. 6: 269–274 (1954).

Presented at the meeting of the New England–Maine–New Hampshire Sections of TAPPI, held in Portsmouth, N. H., June 10–11, 1961.

Justification of Pulp and Paper Mill Instrumentation to Top Management

James E. Overall

In some cases management is unaware of the savings and financial returns that can come from good instrumentation, correctly applied. In approaching the problem, the progressive steps could be as follows. (1) conceive an idea (2) sell and define, (3) specify, (4) buy, (5) install, (6) prove, and (7) maintain. These points are developed with emphasis on (2) as it is related to savings of money and the development of a better product. Suggestions are presented on the operation of an instrument department.

The pulp and paper industry in the United States has reached and is presently passing through a stage in the application and appreciation of process instrumentation that might be considered roughly the equivalent of being a teenager.

To draw some analogies, we might say that we have reached a physical stature that is essentially full-grown, and by and large, the ideas and reactions of top management are both mature and informed in a good percentage of the total group, however, in the remainder of the group the ideas and patterns of use and acceptance of instrumentation are relatively immature, and like a good many teenagers, they simply don't realize what they're missing.

Specifically, top management is in some cases largely unaware of the significant savings and the attractive financial return that can come from good instrumentation, correctly applied to the problems of the industry. But for this situation, we cannot properly hold management responsible, for the reason that they have not been told, or in some cases have not been convinced, that instruments are both practical for and necessary to modern pulp and paper making processes. In considering means to correct the situation, we must acknowledge the fact that it is our job to educate top management in the proper use and appreciation of process instrumentation. This is our ax and we must grind it, because no other group is as interested in this project as are we, and no other group is as qualified as we are to do this job.

In approaching this problem, we may as well face up at once to the fact that our approach must necessarily be built on a financial basis, for the overwhelming reason that every company in the pulp and paper industry is engaged in making money, not paper. Those of us who are intimately associated with the engineering aspects of our profession sometimes

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tend to lose sight of this essential fact; top management, on the other hand, lives with this reality day and night, and this is the language that they understand, and that we must use in our approach.

In coming to grips with this problem, there is a definite sequence of events that we should note. One method of breaking them down might be as follows: (1) conceive, (2) sell and define, (3) specify, (4) buy, (5) install, (6) prove, and (7) maintain.

Let us now touch briefly on these seven points, and then concentrate on the second one, with which we are primarily concerned here. These points, or steps, are those that we must normally take in bringing into being a successful new instrument application.

First, it is necessary that we *conceive* an idea, then a system, by which some problem of production may be solved, or by which some necessary new process may be made possible, practical, and effective. We must, in short, convince ourselves that we have something worth while, and then establish a means of practical accomplishment.

Second, we must then *sell* this concept to our top management, and having sold them on the basic pattern, then *define* the limits of the proposed project. It is necessary that this definition of limits be established at this point, in order that we may fully cover the essential hardware and accessories, while limiting the size of the project to practical budgetary consideration for the period to be considered.

Having sold and defined our idea, we are then in a position to work with the instrument department to *specify* completely the instrumentation that will be required for the project. This specification of the hardware clarifies in our own mind what we need, and permits us to describe to prospective vendors what we will require.

Having set up and distributed the specifications, and having received quotations from qualified vendors, we are then in a position to recommend to the purchasing department that a particular offering be *bought*. It should be borne in mind here that it is our responsibility to determine that the various offerings do meet the specifications, not only as we have written them, but as we conceive the needs of the total project. This is our proper function, and is not one that we can or should thrust onto the purchasing group.

When the material has been received through the purchasing department, it is then the function of the instrument group to *install* all components in a proper manner for carrying out the functions of the project as conceived. This phase includes the physical check-out of individual instruments, and of control loops having a specified function.

When the installation has been completed, and has been certified as ready for operation, it is a continuing function of the instrument group to *prove*, in cooperation with the technical and operating groups, that the system is capable of accomplishing the function originally intended. This is an extremely critical and important phase, and we must never forget that a primary responsibility here is the selling of the idea to all operating personnel who may be associated with the project, because it is they who must operate it in the years to come, and it is they who must ultimately be satisfied with the practicality of the system. We too often convince ourselves that all is as we want it to be, but fail to continue our selling job until the operators are equally convinced. In practical terms, our job is not done until the operators are completely convinced that it is to their advantage to put the equipment on automatic operation, and leave it that way under all but emergency conditions.

Having accomplished the above steps, and having successfully placed in permanent operation a useful system, it is then a continuing responsibility of the instrument group to *maintain* this system in a completely reliable and satisfactory condition at all times. You will know that they have succeeded when the operators call the instrument shop promptly to complain as soon as any malfunction develops; if the operators go on manual control without calling the shop, then you have to some degree failed in your proper responsibility.

Let us return now to our point number two, and deal with the practical aspects of selling these services, and these ideas, to top management, remembering that unless and until we can legitimately show a financial benefit, we have no valid claim upon their time. As a starting point, let us consider the following topics as they relate to the saving of money, or the creation of additional income.

1. By *reducing* use of *expensive components* of our final product. Typically, we might consider here a ratio blending system for the control of use of pulps, such as groundwood, semichemical pulp, sulfite, and broke. With some of these pulps valued at $50.00 a ton, and others at $100.00 a ton or more, it is obvious that if we can use the maximum quantity of cheap pulp and a minimum quantity of expensive pulp, while maintaining a completely safe level of quality, then we can reduce the cost of our finished product.

2. By *reducing manpower* requirements; usually, this means reducing manpower requirements at a later date, as it is extremely poor practice to attempt to justify instrumentation on the basis of immediate elimination of manpower. It is far better to use instrumentation to distribute the actual work done by personnel over a greater area, and is best done at times of expansion, thus utilizing existing manpower to do additional work, and eliminating the need for the hiring of more people. A second point to consider and remember is that we may very well reduce the demand for high-caliber manpower, where decision-making is a prime requisite, by the application of instruments to perform relatively complex functions, such as control of high-pressure, multifuel steam generating equipment. Another such situation might arise in the handling of broke, where batching, sequencing, or continuous process control equipment may eliminate the need for any labor other than that required to load material into hoppers or other containers.

3. By *increasing production;* this is quite obvious, but is more easily said than done. However, two ready examples might be the increasing of machine tonnage by means of a more efficient control of drier drainage through automatic systems, or the production of more pounds of steam through effective automatic control of air and fuel flow to a steam generating boiler. Basically, we refer here to the increase of production from the existing equipment, through no investment other than automatic control equipment.

4. By *reduction of product waste.* This perhaps is most easily illustrated by the reduction of broke through better basis weight control, better moisture control, better color matching and maintenance, better finish through better calender control, and the like. These and many similar functions are demonstrably improved by the proper application of process controls. Every ton of production thus saved is available for sale, and is properly to be credited to better automatic control.

5. By *reduction of capital equipment costs.* Here, we might take as examples the reduction in need for additional large tanks or chests, or the reduction in need for containing or supporting steel and concrete, such as might be accomplished by the use of pipeline type flowmeters, consistency regulators, and particularly blending and batching systems. These savings can be of great magnitude, and it should be remembered here that savings also can be claimed and proved in the reduction of space requirements, since space is an item of definite cost, in both old and new mills.

6. By the *reduction of fuel costs;* this topic is important enough to be listed as a separate item, although it has been used as an example previously. Fuel costs are a major item of basic plant operating costs, and fractions of a cent saved per B.t.u. amount to quite appreciable sums in a year's time. With the current trend to larger and more complex boilers, this avenue becomes extremely attractive.

7. *By reduction of raw material consumption.* Typically,

here we might consider the savings in alum, size, clay, titanium dioxide, and similar purchased raw materials; the savings in these materials become a reality as soon as we are able to meter the materials accurately, and to limit the rate of addition to that which is required as a minimum for proper quality; otherwise, these materials in almost every case are added in excess, in order to avoid occasional production of sub-standard product. Here again, the savings of fractions of a cent per ton will amount to a sufficient sum in a relatively short time to pay completely for the required instrumentation to do the job.

8. By *reduction in the use of water*, or in *reducing the waste of water*. This is becoming increasingly important in all areas of the country, and is accomplished by the metering of water at every important point of use, and point of discharge from the mill. Metering in these cases can be in a relatively crude manner, with accuracy in the order of ± 5% being quite acceptable. Unless you have been involved in work of this sort, it will be difficult for you to conceive of the amounts of money that can be saved by proper use, and particularly by proper reuse, of the available water supply. Similarly, the treatment of selected waste water is becoming attractive, and in many states, is becoming mandatory. By the proper application of instrumentation, such water treatment can be economically accomplished. One of the important fringe benefits of a water-accounting program is the pin-pointing of previously unidentified sources of tremendous waste or abuse of available water. Instrumentation is particularly useful in compelling various mill departments to account for their consumption, rather than arbitrarily assigning all suspected wasted gallonage to other departments.

9. By the *manufacture of better products;* this topic might be considered a portion of item four above, but the meaning here is extended to include quality control measures which are effective enough to warrant a better price for the product, or to warrant the development of completely new markets for an old product previously not acceptable. This is one of the presently most attractive possibilities, and one that is being rapidly forced upon all mill management by the highly competitive situation in which the industry finds itself.

Referring now to the points enumerated above, as a group, it will be well to consider how we actually can best describe to management the benefits to be obtained from these measures. The most attractive method, and the one most easily used, is to determine in each case the actual amount of money that can be saved per ton of product, whether this be a fraction of a cent, or a larger amount. This amount, multiplied by the annual tonnage of the piece of equipment in question, then gives a figure that can be expected annually to be applied toward paying the cost of the instrument package. An alternate approach is to determine the number of pounds of raw material that can be saved per ton of product by use of instrumentation; the annual tonnage as a multiplier again produces a figure, which when multiplied by the cost of the raw material, yields a number of dollars that can properly be claimed. Unless you have done a few of these calculations, you will be surprised by the figures that you obtain, as they will in most cases be considerably more than you had expected. This is probably the best point to state that the optimum pay-out time for a system of instrumentation is usually considered to be 2 years; in other words, the savings, or the gain, attributable to the instrumentation should equal the cost of the instrumentation in that period of time. Obviously, the savings or gain beyond this 2-year period then become clear profit for the remaining life of the instrumentation, which is in the order of ten years in most cases. A shorter pay-out is obviously to be desired, and is frequently accomplished, but in making your estimates it is suggested that you be conservative, as no one will be unhappy if your system is better than you predict, but if it is not as good, then you are placed in a poor position for further personal and departmental growth.

It is worth pointing out once again, and emphasizing, that in properly evaluating the services and contribution of an instrument department, the savings noted above are annually repetitive over a period of many years, so that the worth of the department to the company can actually be established in dollars and cents on an annual basis, by tabulating the results of new applications for the past year plus continuing results from applications in earlier years. A figure that is relatively easy to prove is that an instrument department, including salaries of supervisory personnel, can be expected to return to the company at least five times, and usually ten times, the total amount invested in the department, on an annual basis. This is true even in the light of a point that I will make later, relative to adequate salaries and wages for instrumentation personnel.

If we may consider this portion of the discussion closed for the moment, it will be worth our while to proceed to several practical aspects of running a good instrument department.

It should go without saying that any or all of the job of selling mentioned earlier will be made easier if the department is genuinely popular with top management, with the engineering groups, with the technical groups, and most of all, with the operating groups. As in most situations encountered in life, the best possible approach here is that "nothing succeeds like success." Fortunately, the accomplishment of this goal is relatively easy, if the head of the instrument department will make it a matter of policy, and a matter of common knowledge, that he is ready, willing and able at all times to cooperate with management, engineering, operations, or the technical group, in putting in a temporary installation to prove, disprove, or to evaluate any pet theory or project that one of the groups may originate.

Always, the bugaboo of any fledgling idea is the lack of money with which to carry out the essential physical requirements; the instrument department is in an ideal position to be the activating force in these cases, by having on the shelf in the shop, ready to go, one or more spare recorders for temperature, pressure, flow, pH, ORP, and as many other physical functions as can be managed. Now before all of you depart in a rush for the door, don't despair that this can be done, and can be done in a simple and practical manner. The method is to salvage every old or unused instrument in the mill, place it in some sort of reasonable working order, and then wait for a call for its use. When an application is proposed by a responsible person in one of the critical groups enumerated, enlist the support of the millwrights, pipefitters, welders, and electricians in setting up a basic form of the proposed project, in cooperation with the operating personnel and any other group involved. Once the project has been placed in operation, do everything in your power to guarantee its success, noting all of the factors suggested earlier. Now, and most important of all, as soon as the project is in successful operation, go to the one who proposed it, and lay before him a requisition form made out for a new duplicate instrument, or instruments, to replace those you have let them use temporarily. Virtually every recognized department in the mill has a modest contingency budget which can be utilized for small unexpected expenditures, and it is from this budget that your replacement instrument will now come. You will in almost every case get this new instrument, rather than remove the old one from the satisfactorily operating system, believe it or not. As soon as the new instrument is received, place it on the shelf in readiness for the next project, keeping in mind the fact that where you may have supplied only a recorder in the first place, it is one of the ground rules of the game that you order a replacement recorder controller and always, a new and slightly larger valve than the one that was supplied for the temporary project. Thus, all is well, and everyone is happy, including the instrument department.

Since one of the major stumbling blocks to this approach is the availability of a suitable valve, it is obviously a basic point that every unused control valve in the mill should be in the possession of the instrument department, and that it be

refurbished to a degree that will permit its use in these projects. Once the valve is in operation, the replacement of any borderline parts of the valve or of the positioner is a relatively easy matter, as operating budgets are considerably bigger and more flexible than capital budgets. The general statement here is that you are spending "30¢ dollars" rather than "100¢ dollars," income taxes being what they are these days.

A corrollary of the above approach is that the instrument department should maintain close surveillance on all instruments that are in borderline use, or have slipped into disuse. These instruments should be removed from service unobtrusively, and should be placed in the kitty for special projects, or should be transferred to applications in the same department where they can do a better job. It is important to keep in mind here that transfer of instruments from one department to another is not legitimate, and will make more enemies than the transfer warrants. It is possible, however, to arrange swaps between departments, where such shifts would mutually benefit these departments. Helping department heads to get better service from all of their instruments is a sure-fire way to make your future and that of the instrument department both pleasant and profitable.

In carrying out the program just outlined, it is worth your constant attention and effort to make your instrument shop, your instrument storeroom, and your instrument office real showplaces, and I mean this literally—visitors should always be welcome, and mill management should be encouraged to route all important visitors through this area. This means battling at every opportunity for a better and larger and cleaner location, for more shop test equipment, for adequate personnel safety equipment, for immaculate housekeeping, and for full recognition of the instrument group as a separate organization apart from any group that tends to subordinate it.

As a final point, and one that is worthy of your full attention, I should like to dwell for a moment on the fact that with very few exceptions, the instrument man, the instrument mechanic, the instrument technician, or whatever he may be called in your mill, is the cream of the crop of the entire maintenance force. As many of us realize, but as top management sometimes does not, this man, wherever we find him, must know his job in its every possible ramification, plus having a better knowledge of any process in the mill than does the operator of the process. I think that your instrument supervisor would agree that more than 75% of his service calls turn out to be a situation wherein the operator notices that the recorder is showing something out of the ordinary, and calls the instrument man to "fix the recorder." When the instrument man arrives on the scene, he has a choice of fiddling away a couple of hours on a perfectly satisfactory instrument, or of trouble-shooting the operation itself in order to convince the operator that the recorder is simply reporting that somewhere a valve is closed, a pump is leaking, an obstruction is in the line, the seal water is turned off, or a tank has run dry or is running over. Having found the trouble, and having used all his persuasive powers to convince the operator that he should correct it, the instrument man then returns to the shop to do his own work while awaiting the next such trouble call. Now, my contention is that for these services, and for this degree of judgment and responsibility, the instrument man should be set completely apart from all other maintenance personnel, and should be paid a premium rate for his work, above that of any other operating craft. I deeply and sincerely believe this, and I think that it is a major responsibility that we do everything possible at every opportunity to bring this situation to the attention of top management, in order that we may at the earliest possible time justify and bring about this special recognition of the status of the instrument mechanic, and as an obvious parallel, full recognition and adequate compensation for instrument engineers and instrument supervisors.

Presented at the meeting of the New England—Maine, New Hampshire Sections of TAPPI held in Portsmouth, N. H., June 10–11, 1961.

Continuous Freeness Measurement and Control

G. L. Harman

A description is given of the Bailey continuous freeness recorder and data are given on its operation. Indications are that it provides a reliable instrument for freeness control.

THE need for a continuous measurement of freeness has become more and more evident with modern refining methods and higher speed machines. As continuous stock blending systems increase in number, close control of freeness for each stock becomes more of a necessity along with flow ratio and consistency. Much of the original test work has been described by G. A. Chedomir (*1*), to whom the author is indebted for some of the curves presented.

PRINCIPLE OF OPERATION

As a review of the principle of operation, reference should be made to Fig. 1. The stock sample at controlled consistency and temperature is pumped into the measuring tank. Here a revolving cylinder is rotated at constant speed. This

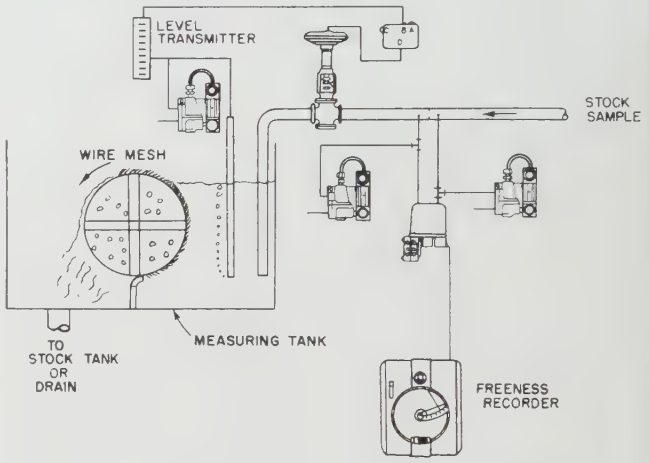

Fig. 1. Bailey continuous freeness recorder

cylinder is sealed on the side and bottom so that any water drained from the stock must pass through the wire on the surface of the cylinder. The drum is actually a "dandy" roll and the system thus operates similar to a rotary filter or decker.

As the cylinder rotates, a film of stock quickly forms on the wire and conditions similar to a wire on a fourdrinier machine are simulated.

The level ahead of the cylinder is measured and controlled by the valve in the stock sample line. The flow in the sample line thus varies with the amount of water passed through the film of stock and wire. The measurement of this flow is used as the index of freeness.

FACTORS AFFECTING DRAINAGE

As you well know, temperature, consistency, and freeness all affect the drainage rate on the unit shown in Fig. 1. Tests were made under controlled conditions to determine the effect of each one of these measurements.

Figure 2 shows a plot of gallons per minute versus temperature with the freeness and consistency held constant. The curves show various rotor speeds and illustrate the effect of temperature. If temperature is held constant, no correction need be made to the freeness measurement; and if tempera-

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ture varies, a correction can be made from the chart record or done automatically through calibrating relays. It should be noted that these are straight-line curves with reference to gallons per minute rather than the variable correction factors applied to the Canadian freeness tester.

Figure 3 is a plot of gallons per minute versus consistency

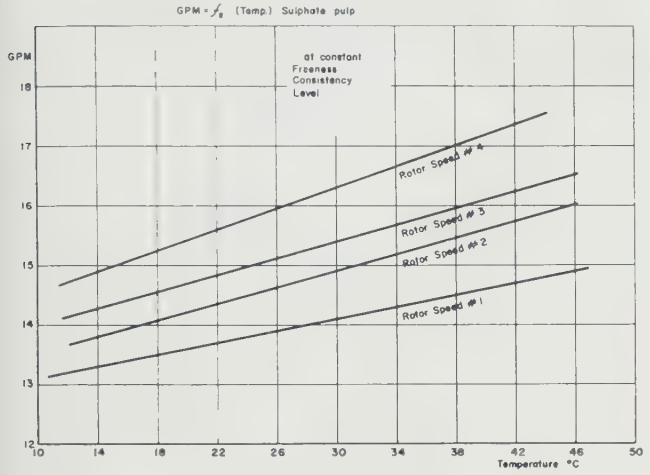

Fig. 2. Pulp temperature versus gallons per minute

with freeness and temperature held constant and curves for each drum speed were again made. From these curves, you will note that if consistency is held within 5% of the reading, we will be within the limits of laboratory testing.

Figure 4 is a plot of gallons per minute versus freeness with consistency and temperature held constant and again the various drum speeds are shown. From these curves and the previous ones on consistency, it will be seen that freeness

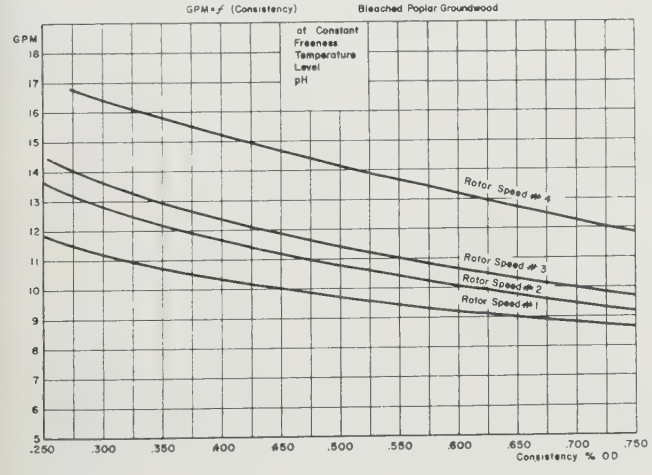

Fig. 3. Consistency versus gallons per minute

affects the drainage rate approximately twice as much as consistency.

TESTS ON PRODUCTION WORK

From these laboratory tests, a production unit was built in order to conduct further plant and operating tests. Our first tests on this unit involved were made at the Research & Development Center of Kimberly-Clark Corp. A pulper with enough capacity for half-hour runs was used and the freeness varied by varying the amounts of groundwood and other stocks. Figure 5 illustrates one of the curves made on the production unit and it is seen that a nice calibration can be obtained under these controlled conditions.

FIELD TEST WORK

The unit was then shipped to an actual plant installation for continuous operating experience and here several sampling systems were reviewed. The first installation involved actually controlling consistency from a consistency controller so that the stock sample was diluted down to a standard con-

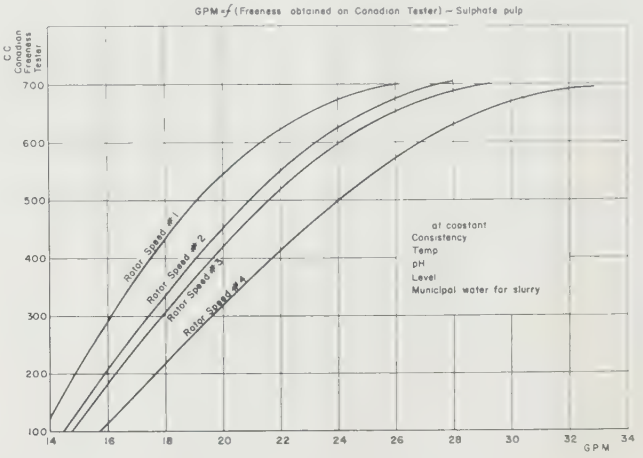

Fig. 4. Gallons per minute versus pulp freeness

dition of approximately 1% consistency for use in the freeness tester. These tests were made on semichemical stock. It was thought that if we had controlled consistency at the refiner, the consistency in the tank could be controlled by a simple ratioing system and avoid the expense of the consistency regulator. Figure 6 illustrates the type of sampling system incorporating this feature.

Here a sample line is taken off at the refiner at a controlled consistency. Downstream from the metering pump the stock was diluted with water through a flow regulator to bring the

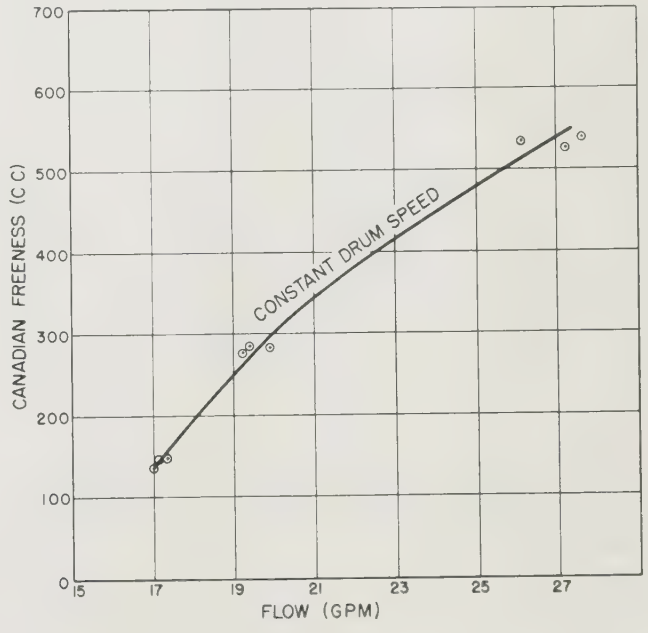

Fig. 5

consistency down to about 0.75%. It should be noted that the level control valve is now in a by-pass line around the pump so that we would not dead-end the pump under any circumstances.

The foregoing sampling system worked very satisfactorily but later tests on a high freeness, long fiber stock caused plug-

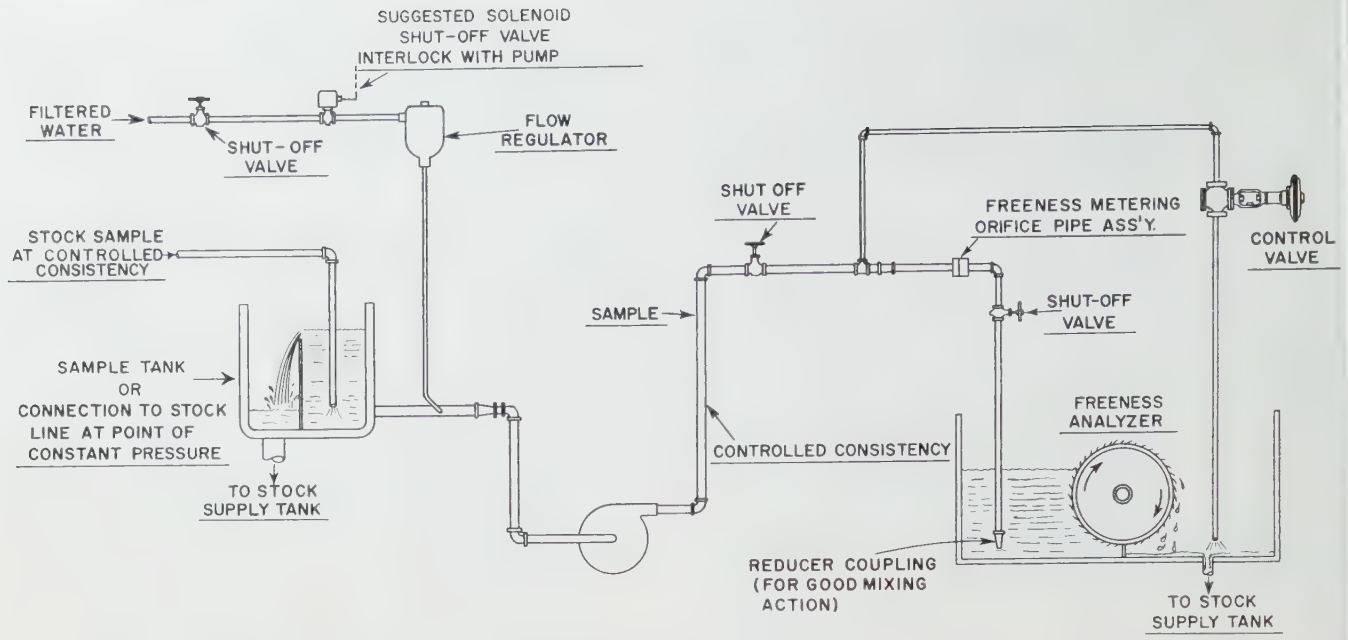

Fig. 6. Continuous freeness recorder piping arrangement illustrating consistency control by flow ratio

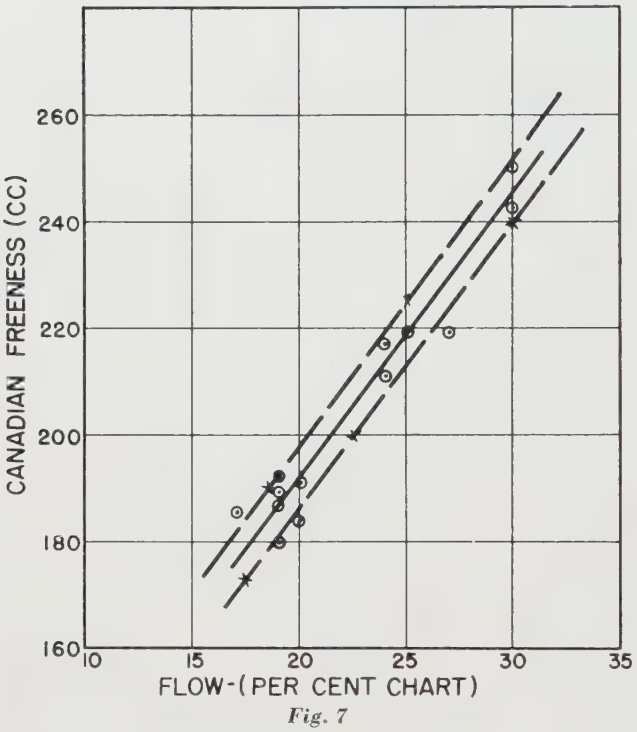

Fig. 7

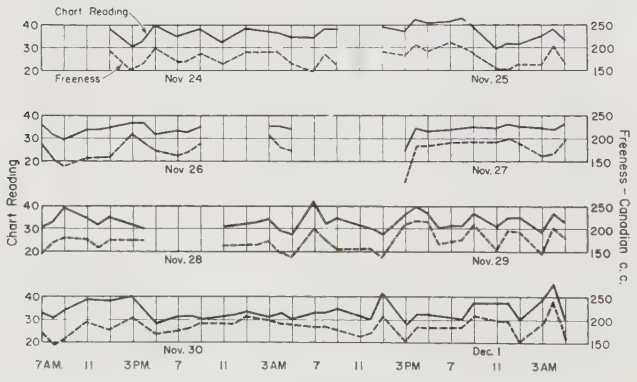

Fig. 8. Freeness and the continuous freeness tester chart reading for the period of Nov. 24 to Dec. 1, 1959

ging problems at the pump suction. The pump could not handle a high freeness stock at a consistency of 4%. The dilution water was then brought into the pump suction as shown in Fig. 6. It is also necessary when using this system to have the sample tank or point of constant pressure at the pump suction. This then provides constant sample pump operation. This drawing also illustrates that the sample line going into the vat is reduced down by a coupling to give a jet action and consequent good mixing action in this headbox.

PLANT TESTS

Numerous tests were conducted in actual plant operation and Fig. 7 is a plot of some of the points made during portion of these tests. The dotted lines shown on this curve give the ± 5 tolerances. It should be noted that most of the points fall within these limits.

It was now desired to operate on a continuous basis and see how the calibration held up over periods of time. Figure 8 is a

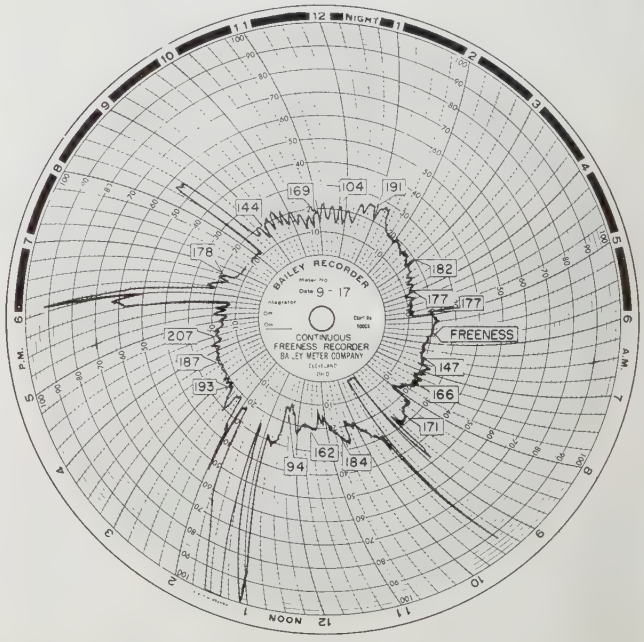

Fig. 9. Freeness under manual control. Periodically, samples of pulp were drawn and freeness tests run to compare with chart record

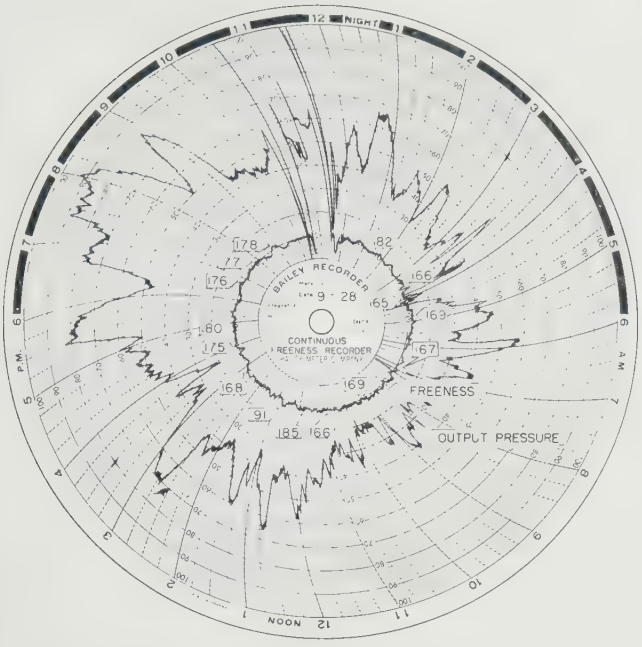

Fig. 10. Freeness under automatic control. Periodically, samples of pulp were drawn and freeness tests run to compare with chart record

plot of the chart reading and laboratory freeness samples taken from a period of Nov. 24 to Dec. 1, 1959. The curves are deliberately on different ordinates as we were trying to show whether or not the curves followed each other in the proper pattern. These plots seem to indicate the continuity of calibration and that the device could be used for either manual or automatic adjustment of refiners.

Figure 9 is a chart showing the freeness record under manual control and this was operated in this fashion for several weeks.

As the refiner and recorder were set up for automatic control, the response of the system and controllability were then studied under automatic control conditions. The controller would send out a 3 to 15 lb. signal which was applied to a pneumatic to hydraulic valve in the refiner control system. Limits were, of course, set on this system but the control could operate between approximately 900 and 400 lb. refiner closing pressure.

Figure 10 is a chart taken showing automatic control of freeness and here two records are illustrated. The inner record is the freeness measurement while the outer record shows the control pressure going to the pneumatic to hydraulic system calibrated so that 100% of chart equals 900 lb. closing pressure and 0% of chart equals approximately 400 lb. closing pressure. The refiner was the third of three refiners in series and the only one under automatic control. It should be noted that the normal recorder chart for this unit would have four records when controlling a refiner. Two of the records are shown on this chart; the other two would be temperature and vat level in the freeness analyzer. Temperature, of course, is recorded to correct the reading if necessary and level to show if this part of the system is working correctly.

It should also be noted that when diluting stock with water in the ratio of about 10 to 1, the temperature will be affected more by water temperature than stock temperature. It has been found to date that this is not too great a variable in our readings.

Figure 11 illustrates the actual unit in operation showing the pulp mat and other features.

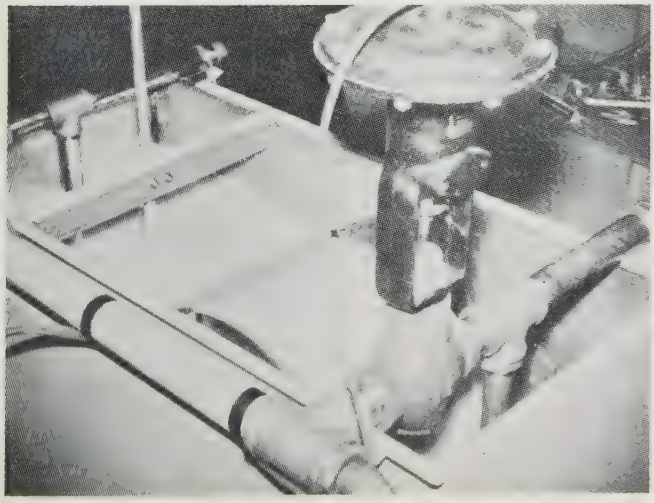

Fig. 11. Bailey continuous freeness measuring tank

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SUMMARY

The laboratory test work as well as the tests under actual plant conditions indicate that this has been a reliable measurement for the control of freeness. Operators have been instructed to clean the unit once a week and because of its reliability, and low maintenance the operators have used this as an added tool in their stock preparation systems. Further field test work is continually being done and efforts will be made to keep these data published.

LITERATURE CITED

1. Chedomir, George A., "Continuous Freeness Recording Controller," Fifteenth Engineering Conference of the Technical Association of the Pulp and Paper Industry, Jacksonville, Fla., Oct. 24–28, 1960.

Presented at the meeting of the New England Maine-New Hampshire Sections of TAPPI held in Portsmouth, N. H., June 10–11, 1961.

Methods for Studying Resin Distribution*

CHARLES J. SEILER

Project No. 765 of the Paper Laminates Committee was set up to determine the availability of quantitative or semiquantitative methods for measuring the distribution of various resins in cellulose materials. The survey is divided into four major sections: microscopy extraction, physical analysis, chemical analysis, and autoradiography.

PROJECT No. 765 has been set up by the Paper Laminates Committee of TAPPI to determine the availability of quantitative or semiquantitative methods for measuring the distribution of various resins in cellulosic materials. Although primarily concerned with the thermosetting saturants used by the laminating industry, many of the techniques may have utility for studying the distribution of thermoplastic coatings and impregnants.

The demands being made of molded and laminated products are increasing rapidly, and we cannot expect to satisfy them by trial-and-error methods of research. We must attain a far better understanding of the precise physical and chemical interrelationships between resin and paper in an impregnated product if we are to compete more effectively with new developments in competitive products. The need for test methods for resin distribution is only one facet of this problem.

The rate and extent to which a resin system penetrates into the fiber structure of paper or cloth is a function of many resin variables such as viscosity, polarity, interfacial tension, molecular weight, solvent type, temperature (*1*), and pressure, as well as a large number of paper variables such as degree of hydration, fiber length void concentration, and density.

One of the major factors affecting the degree to which a given resin penetrates into the fiber structure of the paper is the solvent employed to carry it. Water is, of course, one of the best and methanol close to it in swelling action on cellulose. This, in turn, opens the way for the resin to enter the cell walls and capillaries. Some of the chemicals which are reported to swell cellulosic materials to a greater extent than water are formic acid, formamide, the lower primary amines, and pyridine (*2*). Differences in swelling power of a resin-solvent system result primarily from differences in polarity. Swelling coefficient can also be related to a tendency to form hydrogen bonds with the hydroxyls of cellulose. Molecular size may also be an important factor. It has been reported that the intermicellular spaces in paper are in the order of 10 to 1000 A., so that for optimum impregnation the resin molecule should not be greater than 10 times the size of the monomer molecule (*3*). Although nonpolar systems do not penetrate fibers themselves, they can replace polar liquids in which they are soluble.

Landells reports some effects of degree of impregnation of melamine resins into fibers of cotton, rayon, and wool on the physical properties of the system (*4*).

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* Progress Report—Project No. 765, TAPPI Plastics Laminates Committee.

Figure 1 illustrates some of the factors affecting paper saturability (*5*). Note the marked differences between the saturation levels for hydrocarbon oil and polar test solution (alcohol-solvated phenolic resin). This can be attributed to the fact that the oil is primarily filling the voids and pores and is not being absorbed into the fibers. This was further borne out by penetrability tests (Williams penetrometer) during which the oil penetrated rapidly through the paper, indicating little absorption into the fibers with consequent fiber swelling to impede the progress of the oil.

A surprisingly large difference in saturability was apparent for two supposedly similar papers obtained from different suppliers. Paper C absorbed considerably more resin than paper A indicating that it may have low compactness, high void, and pore concentration, or highly beaten fibers.

One of the reasons for the lack of availability of standard techniques for measuring resin distribution stems from the fact that most experimental methods employ a combination of microsectioning, microscopic, and staining techniques usually coupled with microphotography, and this has somewhat dampened incipient enthusiasm. Much of the experimental work that has been reported has been done by either nonprofit organizations such as universities and paper institutes, or in Europe by the various textile and dye producers. Germany has perhaps published more than all others in the field of dye and resin distribution, motivated probably by their

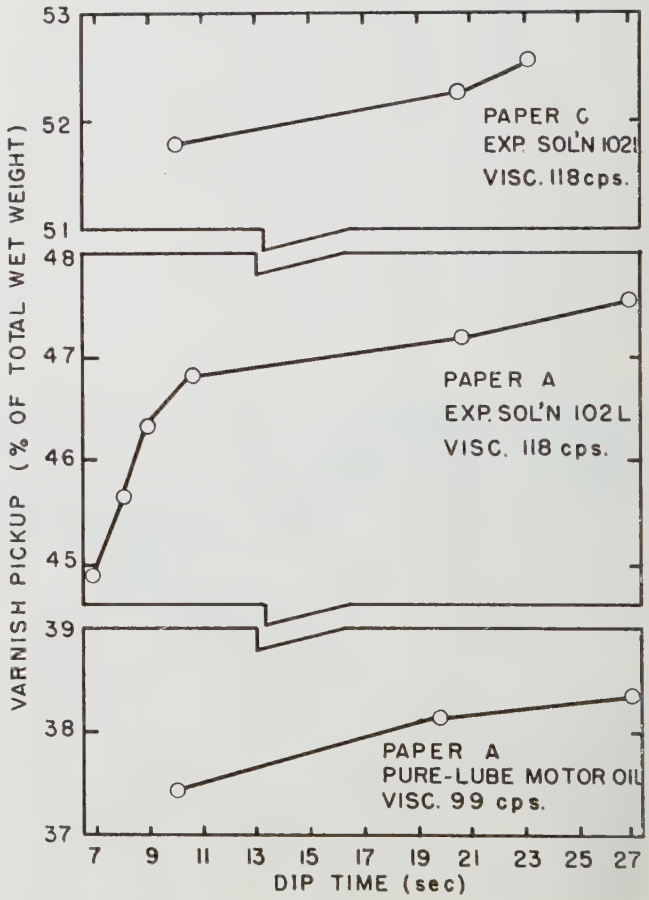

Fig. 1. Effect of paper and saturant on total pick up

interest in dyes. In any event, relatively little work in this field has been published by our own paper industry or by paper impregnators.

RESIN DISTRIBUTION TECHNIQUES

The survey has been divided into four major sections: microscopy, extraction–physical analysis, chemical analysis, and autoradiography.

1. Sectioning—Microscopy
   May involve:
   a. Staining
   b. Microphotography
   c. Electron micrography
   d. Special lighting techniques—polarized light, ultraviolet, optical staining
   Attributes:
   1. Semiquantitative
   2. Most descriptive
2. Sectioning—Extraction–Physical Analysis
   May involve:
   a. Gravimetric methods
   b. Spectrophotometric methods
   c. Colorimetric methods
   Attributes:
   1. Simple techniques
   2. Quantitative
3. Sectioning—Chemical Analysis
   Attributes:
   1. Most specific
   2. Simple techniques
4. Sectioning—Autoradiographic Analysis
   Attributes:
   1. Does not alter sample
   2. Difficult and expensive

MICROTOMING METHODS

Before discussing the various methods available it may be wise to spend a moment on the one technique common to all methods—microsectioning (*6, 7, 8*). This may range from a simple picking off of individual fibers, to adroit use of a razor, to elaborate microtoming techniques employing special knives or diamond wheels and sections as thin as 2 μ. However, 10 to 15 μ probably represents the normal limit for thinness of section. Special techniques are required to obtain specimens thinner than this.

An excellent booklet on microscopy was published by American Cyanamid called *Dyestuff Tech. Bull. No. 770* (*9*). Although it emphasizes microscopic techniques, it has a good section on microsectioning. There are also a number of papers published by The Institute of Paper Chemistry at Appleton on microsectioning (*8*). A relatively simple method employing a technique developed by Hardy is called a Callatome. This was designed for longitudinal sectioning and is quite simple and inexpensive. They claim sections down to 5 to 10 μ. The Hardy method for cross-sectioning fiber or paper is outlined in Fig. 2 (*6*). The method is the basis for several commercial microtomes.

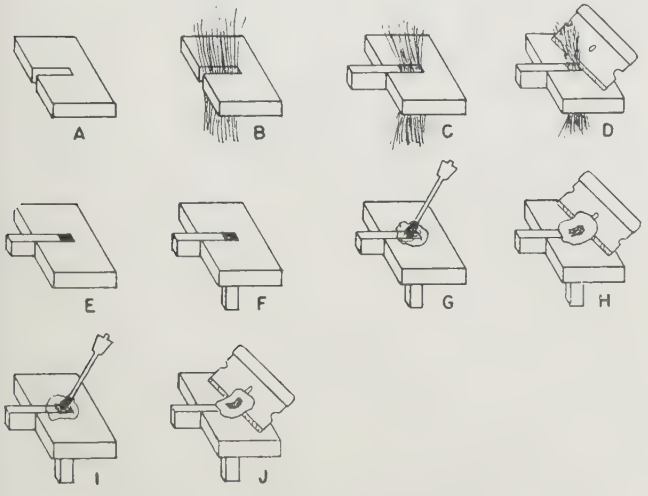

Fig. 2. Hardy method of microtoming

The sequence in the Hardy Technique is as follows: (1) material or fibers are inserted in the slot, (2) cut off evenly on both sides of plate, (3) plunger pushes fiber out top of slot, (4) cellulose acetate in acetone or a collodian solution is brushed over top of fibers, (5) fibers cut to produce flat section, (6) operation (4) and (5) repeated after raising fibers by known amount. The section is then mounted on a microscope slide in a permanent mounting medium.

Now let's examine in more detail the techniques which have been used to study resin distribution.

METHOD I. SECTIONING—MICROSCOPY—PHOTOMICROGRAPHY

First and foremost under this heading are the staining or dyeing techniques. Two basic methods are available: (1) selectively dyeing the paper or fabric, (2) selectively dyeing the resin either in the soluble form before impregnation or in the cured state after impregnation.

Which of the two methods is employed depends on the resin system used, its state of cure, and the amounts of resin present. For dyeing cellulosics such as paper or cotton, the direct dyes are usually preferred, especially the azo dyes, since they are particularly substantive to cellulose, and conversely, do not dye most cured resin systems (*10*).

Examples of dyes which have worked well are:

Solar Blue G (11)	Pontamine Deep Blue
Flavine R (11)	Calcodur Blue SL (PR-71)
Acid Fuchsin	Sudan III

The azo dyes can be used to estimate concentration as well as distribution of resin in the fiber by comparing differences in color against a color scale. The resin by reducing the ability of the fiber to swell diminishes its affinity for the dye. Therefore, both resin concentration and distribution become functions of color intensity.

However, a more direct method for measuring resin distribution employs acid dyes which do not dye cellulosics. These will react with the basic amino groups in the amine resin (melamine, ureas, etc.) (*6, 12–15*) distributed in the cellulosic fibers in the cured or partially cured state, while they do not affect the fibers themselves. They are particularly appropriate because they can actually be used for determining differences in degree of polymerization of the resin (*11*). In an acid medium, for example, for a given amount of resin in the cellulosic, the coloring increases with degree of polymerization. In an alkaline medium, coloring decreases with degree of polymerization. This results from differences in amount of hydrolysis of the resin occurring under acidic and alkaline conditions.

Examples of useful acid dyes are: (*16*)

Kiton Blue II
Disulphene Blue VNS
Anthraquinone Blue BN (4)
Brilliant Red Sulfone B (11)
Scarlet Sulfon GWL (11)
Ponceau 6R Extra (15:4)

When amino resin systems are being studied, two approaches should be considered. Where quantity of resin present is of primary interest direct dyes work well (either added to resin solution or by dyeing cured product) but where the precise distribution of the resin is the major factor the acid dyes offer more flexibility.

For phenolic systems where no easily reactive groups are present (as in the case of acid dyes with amino resins), it is usually preferable to add the dye to the resin solution prior to impregnation. Here, care must be taken to choose a dye which has similar molecular weight and dielectric properties to the resin to avoid selective absorption of the one or the other. Actually very little has been reported in the technical journals on the staining of phenolic systems. However, the

author thinks that red acid dyes would work best in the resin solution, whereas green, or blue, direct azo dyes would work best on the cured product to selectively stain the cellulose.

Another method of somewhat doubtful value for studying resin distribution involves the use of ammoniacal silver nitrate (Tollens reagent) (*11*). This deposits silver on and in a fiber in the presence of resins made with formaldehyde. However, the aldehyde groups on cellulose will also produce color so the method has limited utility.

Other methods, besides dyes, used to visually gage the distribution of resin are as mentioned previously: (1) electron micrography, (2) optical staining, (3) polarized light, and (4) ultraviolet light.

These will be discussed briefly.

Electron microscopes are useful where large increases in resolution are required (*17, 18*). The individual dye or resin particles on the surface of the fiber can clearly be seen. Its advantage (besides resolution) is that no contaminant is added to the system. Its disadvantages are that it requires special equipment and produces a very limited field of view.

Optical staining employs a technique whereby the sample is colored with different hues of light from beams directed at it from different angles (*9*). The image is produced from a combination of transmitted and reflected light. It is frequently used in conjunction with a staining technique to highlight different areas. It is particularly useful where large differences in refractive index are present in the sample.

It should be mentioned that it is extremely important in viewing a dyed section to employ the proper amount, type and angle of light incidence on the sample. It is frequently this area that is slighted in studying resin distribution.

One further technique in microscopy involves either the use of fluorescent dyes (*15, 19*) or takes advantage of the natural fluorescence of some materials. Melamine, for example, absorbs strongly at 2537A (*18*). It is extremely important in working with ultraviolet microscopes to eliminate all stray visible light. Even at best, the image is frequently not sharp enough to see precise distribution.

A number of photomicrographs were prepared of cloth and paper cross sections and of individual fibers to study the utility of the various staining techniques. Of particular interest was a series showing the differences in penetration of a group of phenolic resins into cotton drills. These resins differed primarily in being either water, alcohol or xylol soluble. Penetrability into the fiber bundles was markedly reduced as the resin progressed toward the xylol soluble stage. Dyes employed were Fast Acid Red B, Rhodamine B (Basic) and Calco Oil Red. The latter was chosen because of its solubility in xylol. All dyes were added to the resin solution prior to impregnation and drying.

The distribution of a water dispersible urea resin in a bleached sulfite paper was vividly illustrated by dyeing the impregnated and cured paper with aqueous Calcocid Brilliant Blue FFR (acid type). This showed that the urea resin was distributed primarily as discrete particles clinging to the outside of the fiber bundles; little or none of the resin having penetrated into the bundles.

Photomicrographs were either 100X or 230X magnification. A definite limit to magnification results from the excessive loss in depth of focus which occurs at higher magnifications.

METHOD II. SECTIONING—EXTRACTION—PHYSICAL ANALYSIS

The second general technique listed previously involves the use of physical methods for removing the resin from the microsections, followed by simple gravimetric analysis, or perhaps by spectrophotometric or colorimetric analysis. The method most frequently involves longitudinal sections and is concerned with the determination of the depth of penetration of a resin system into a substrate.

An example of the use of the colorimetric technique is given by Browning and Isenberg (*20*) who dissolved out a starch impregnant from 15 μ thick longitudinal paper sections and determined starch concentration by colorimetric methods against standard solutions (see Fig. 3).

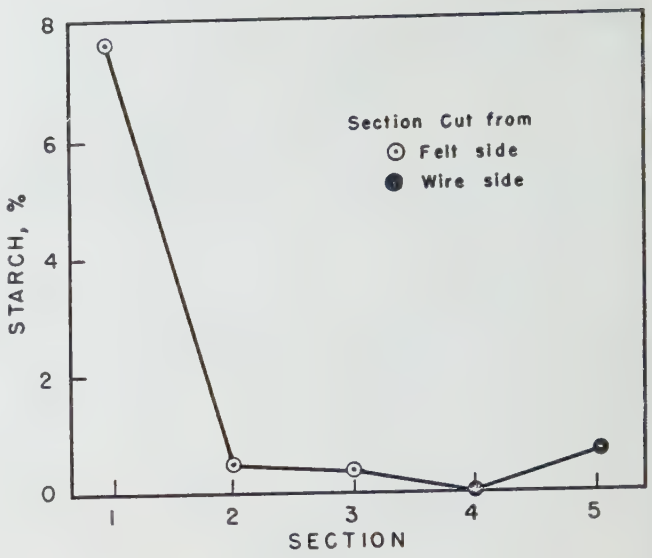

Fig. 3. Starch distribution in paper

Melamine distribution can be determined by spectrophotometric methods since it absorbs very strongly at 235 mμ (*21*). The technique is to extract the melamine with hydrochloric acid and the absorptivity of the solution is measured and compared with standards. This is a quick and fairly reliable method for melamine and probably can be used for other resin systems.

There are a number of solvent systems which have been used to extract both cured and uncured resin from paper (*22*). Uncured resin is no problem but as the resin advances in cure, better solvents for the resin system are required.

For phenolic systems cyclohexanone (*23*), *b*-naphthol (*24*), and dimethyl formamide (*1, 22*) are probably the best; while benzylamine is good for ureas. The author would caution against the use of acetone as an extraction solvent since work at Behr-Manning (*25*) has shown that it reacts to an appreciable extent with phenolic and other resins containing methylol groups or free formaldehyde and gives erroneous results.

METHOD III. SECTIONING—CHEMICAL ANALYSIS

The third classification mentioned employs microsectioning as before but determines the amount of resin present in the microsections by chemical methods based on the reactivity of the chemical groups present in the resin structure. This method is particularly useful for quantitative detection of nitrogen containing resins (*26, 27*) (melamine, ureas, polyamides, nylon, etc.) since simple Kjeldahl nitrogen can be run on the microsections. Concentrations of resins containing reacted formaldehyde can be determined from measurement of total formaldehyde present (as CH_2OH, CH_2O, CH_2OCH_2). Resins containing sulfur, such as thioureas, are detected quantitatively by either sulfur analysis using a Paar bomb (*26*) or by ultraviolet absorption techniques such as mentioned earlier. A rapid reliable method for quantitative analysis of amine resin involves the use of chromotropic acid (*26*) which reacts readily with these resins to form a clear solution. Colorimetric measurements are then made on the solutions.

METHOD IV. SECTIONING—AUTORADIOGRAPHIC ANALYSIS

Another precise method for determining resin distribution which does not fall into any one of the three categories discussed previously involves the use of radioactive sulfur (*29*).

This is incorporated into a urea resin solution in the form of radioactive sodium sulfite before the resin is applied to the paper and cured. Microsections of the sheet are then examined either with a Geiger counter, or autoradiographs could be taken of a cross section of the sheet. This gives a rather vivid picture of the resin distribution.

Very comprehensive articles have appeared in *Melliand Textilber* (*29*, *30*) which describe the use of special radioactive techniques for measuring the distribution of C-14 labeled urea and etherified urea resins in cotton and rayon. By reducing the distance between the radioactive element and the radiation sensitive substrate (sensitized plate) a reduction in scatter of radiation is attained and a much sharper image results. This reduces one of the major drawbacks inherent in autoradiographic techniques which results from the fact that a radioactive source emits radiation in all directions which tends to produce a fuzzy image.

It is apparent that there are a variety of techniques available for determining the precise distribution of a resin in and around the cellulosic fibers. Although none have been studied in sufficient detail to warrant consideration as a standard TAPPI procedure, some of them can be used to obtain valuable information concerning resin-fiber distribution.

Future work on Project 765 will involve further evaluation of specific procedures applicable to a given resin system, such as phenolics, to determine if one or more of them can be formalized and recommended as a standard procedure.

LITERATURE CITED

1. Wolfrom and Nuessle, *Textile Res. J.* 22: 246–253 (1952).
2. Stamm, A. J. and Tarkow, H., *J. Phys. and Colloid Chem.* 54: 745 (1950).
3. Nazzaro, R. T., *Tappi* 33, No. 1: 23 (1950).
4. Landells, G., Mhatre, S. H., and Narasimhan, K. I., *J. Textile Inst.* 39: T-148–T-152 (1948).
5. Seiler, C. J., *Tappi* 40, No. 10: 200A (1957).
6. Royer, G. L. and Maresh, C., *J. Soc. Dyers and Colourists* 63: 287–293 (1947).
7. Davis, S. J., *Textile Res. J.* 22: 192–199 (1952).
8. Graff, J. H. and Schlosser, M. A., *Paper Trade J.* 114, No. 8: 119 (Feb. 19, 1942).
9. Calco Tech. Bull. No. 770, American Cyanamid Co., Bound Brook, N. J. (n.d.)
10. Boss, R. A., *Tappi* 42, No. 7: 185A–190A (1959).
11. Mauri, D. F. C., *Ing. textil* (*Barcelona*) 22, No. 112: 34–35, 37, 39–40 (1955).
12. Norwick, B., *Ind. Eng. Chem., Anal. Ed.* 16: 274 (1944).
13. Stafford, R. W., et al., *Paper Trade J.* 120, No. 16: 51–56 (1945).
14. Graff, J. H., *Paper Trade J.* 122, No. 5: 41–43 (1946).
15. May, K., Rothe, O., and May, L., *Melliand Textilber* 40, No. 9: 1069–1073 (1959).
16. Anon., *Textile Ind. and Fibres* 236, July 1952.
17. Powers, D. H., *Ind. Eng. Chem.* 37: 188–193 (Feb. 1945).
18. Landes, C. G., *Tappi* 33, No. 9: 463–471 (1950).
19. Ruile, Hans, *Melliand Textilber* 38: 442 (1957).
20. Browning, B. L. and Isenberg, I. H., *Tappi* 38, No. 10: 602–603 (1955).
21. Hirt, R. C., King, F. T., and Schmitt, R. G., *Anal. Chem.* 26, No. 8: 1273–1274 (1954).
22. Bhargava, R. P., Hoffman, H. A., Stannett, V. T., *Tappi* 38, No. 6: 383–384 (1955).
23. Wallhaüser, H., *Kunststoffe* 49: 171–173 (Apr. 1959).
24. Durez Control Test #278 (rev. June 1952). Knowlton Bros., Watertown, N. Y. (private correspondence).
25. Seiler, C. J. and Morrison, A., unpublished data. Behr-Manning Co., Troy, N. Y.
26. Morath, J. C. and Woods, J. T., *Anal. Chem.* 30, No. 8: 1437–1440 (1958).
27. Swann, M. H. and Esposito, G. G., *Anal. Chem.* 30, No. 1: 107–109 (1958).
28. Hudson, F. L., James, H., and Putman, J. L., *Tappi* 38, No. 9: 166A–169A (1955).
29. Enders, Heinz and Zörkendörfer, R., *Melliand Textilber* 40: 1076–1082 (1959).
30. Enders, Heinz, *Melliand Textilber* 38: 547–555 (1957).

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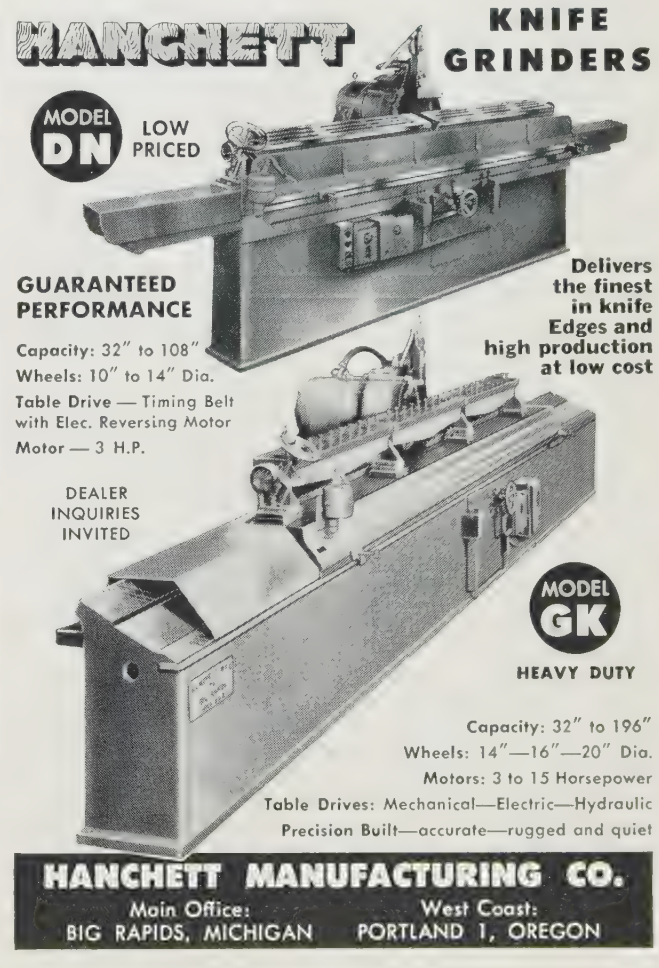

Comparison Between the Modified Emanueli Porosimeter and Other Paper Air Resistance Testers

G. TROGU

The working principles of the air resistance testers (Emanueli, Gurley, and Schoppei) generally used for paper used in cable manufacture are described. A brief description of the modified Emanueli tester is also given. Transformation formulas from paper air resistance in Emanueli units to the corresponding values in Gurley and Schopper units are worked out. In addition, an experimental comparison between Emanueli and Gurley testers has been carried out, both for checking the reproducibility of the methods and reliability of the transformation formula.

It is very important for cable manufacturers to have the possibility of determining the porosity (or its reciprocal form: the impermeability) of the paper used for the insulation of conductors. Porosity is the property that allows a fluid (generally air) to flow through the paper under a certain pressure. This characteristic is very important in cable manufacture as it is a controlling factor in the dielectric strength of the paper itself (*1*). The paper porosity, generally called air resistance, can be determined by several means.

Some methods have been used in the past based on the absorption of liquids by the paper (*1*). However, these methods have been found to give inconsistent results because under the action of the majority of liquids, the cellulose fibers swell or contract, altering in this manner the dimensions of the spaces left between them.

Among the instruments using gases, the most interesting and most used are the Gurley Densometer (*4,5*), the Schopper Porosimeter (*3*) and the Emanueli air resistance tester (*1*). All these instruments give an empirical measure of the air resistance except the Emanueli tester which gives an absolute one.

The purpose of this paper is to describe the modified Emanueli air resistance tester and to define some transformation formula from Emanueli air resistance to that measured with the other instruments mentioned above, and vice versa.

FUNCTIONING PRINCIPLE OF THE EMANUELI TESTER

The principle on which the instrument is based is to compare the paper to be tested with a standard of porosity, provided by a glass capillary tube, as schematically represented in Fig. 1.

From A, a constant flow of air, supplied by a small pump, passes through the sample under test C (having a surface area of S sq. cm.) and a glass capillary tube D. The pressure differences Δp_1, and Δp_2 between the two sides of the paper sample and the capillary, respectively, are measured by the two water manometers M_1 and M_2.

It is supposed that the flow be sufficiently slow to neglect its kinetic energy, and the pressure difference small enough to neglect the variation of volume across the paper thickness, due to the air compressibility.

The volume of air per time unit which flows through the paper under test will then be directly proportional to the pressure difference Δp_1, and to the surface S. The same air flow will be inversely proportional to the paper thickness s, to the air viscosity η, and finally to a coefficient I which represents the resistance of the paper to the air flow. Therefore it follows that:

$$Q = \frac{\Delta p_1}{\eta} \frac{S}{sI} \tag{1}$$

In this equation, if Δp_1 is given in dynes per square centimeter, η in poises, s in centimeters, S in square centimeters, Q in cubic centimeters, per second, I will result in cm^{-2}. The volume per time unit Q that flows through the capillary tube, is related to the capillary characteristics by the Poseuille law:

$$Q = \frac{\Delta p_2}{\eta} \frac{\pi r^4}{8l} \tag{2}$$

where:

r = inner radius of the capillary
l = length of the capillary

It is evident that the volumes per time unit which flow through the paper and the capillary are equal. Therefore:

$$\frac{\Delta p_1}{\eta} \frac{S}{sI} = \frac{\Delta p_2}{\eta} \frac{\pi r^4}{8l}$$

from which

$$I = \frac{\Delta p_1}{\Delta p_2} \frac{S}{s} \frac{8l}{\pi r^4} \tag{3}$$

As the term $8l/\pi r^4$ is a constant for a given capillary, instead of (*3*) it would result that:

$$I = k \frac{\Delta p_1}{\Delta p_2} \frac{S}{s} \tag{3'}$$

in which

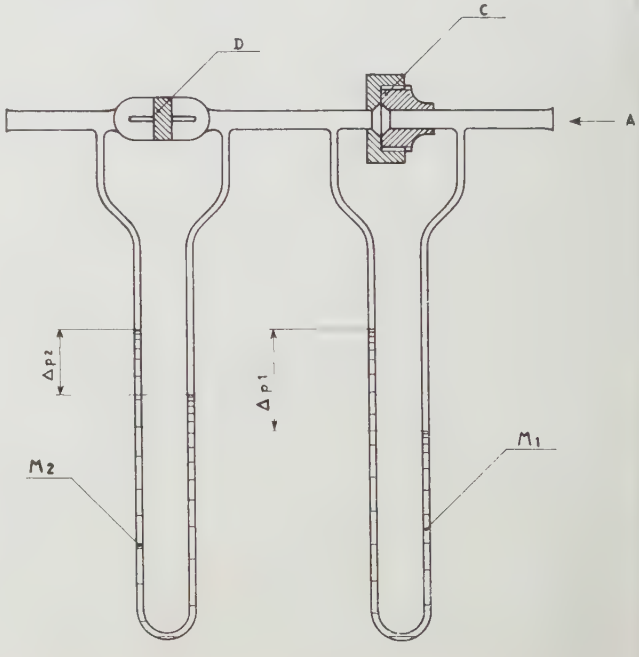

Fig. 1

G. Trogu, Cables Research Laboratories, Pirelli S.p.A., Milan, Italy.

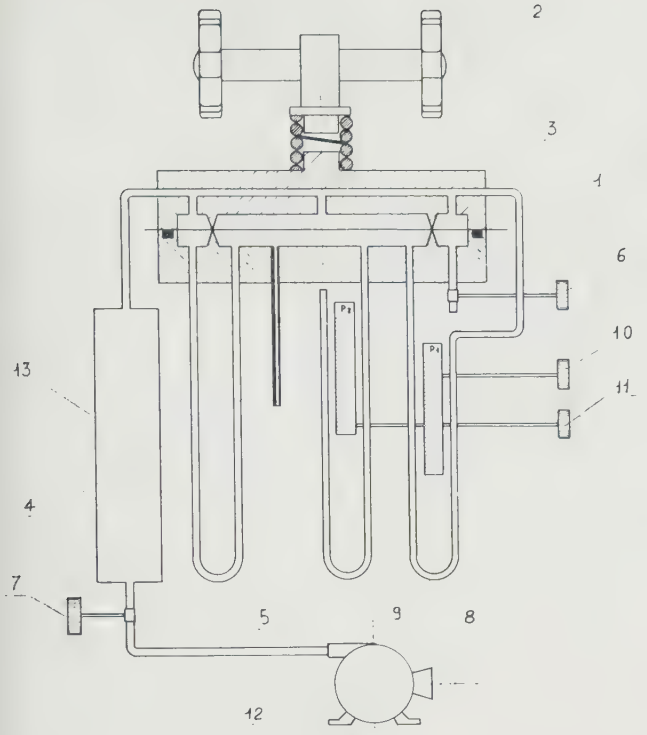

Fig. 2

$$k = \frac{8l}{\pi r^4}$$

As S is also given and is equal to 10 sq. cm., the equation (3′) becomes:

$$I = \frac{k'}{s} \frac{\Delta p_1}{\Delta p_2} \qquad (3'')$$

in which

$$k' = 10k$$

The paper air resistance is then simply determined by the ratio of the pressure differences read from the manometers M_1 and M_2 multiplied by k'/s. Generally it is preferred to use the practical air resistance A instead of I in absolute units. These two sizes are related by the formula:

$$A = I \times 10^{-5} \qquad (4)$$

An important characteristic of the Emanueli method consists in the fact that the same instrument can be used for testing paper with low or high air resistance and the measured values do not depend on the value of the pressure difference, thickness of the paper and air temperature.

Figure 2 represents a schematic drawing of the modified Emanueli air resistance tester* while in Figs. 3 and 4 photographs of the instrument are visible.

In Fig. 2, (1) represents the paper under test on which a rigorously constant mechanical pressure is exerted by means of a spring operated by knobs (2). A guard ring (3) assures that all the flow of air passing through the paper, also flows through capillary (4). The equality of pressure on the paper under test and on the guard ring is checked by means of manometer (5), and, if necessary, adjusted by knob (6), which alters the air flow through the guard ring, while the flow through the paper is adjusted by knob (7). The manometers M_1 and M_2 are marked by figures (8) and (9) respectively. The scales of the manometers can be vertically displaced by means of a track coupling, operated by Knobs (10) and (11). The pump for the circulation of the air (12) and pressure homogenizing tank (13) (that ensures a constant and regular air flow) are also visible in Fig. 2.

* Literature on the modifications of the Emanueli air resistance tester has been published, for example, in (*2*).

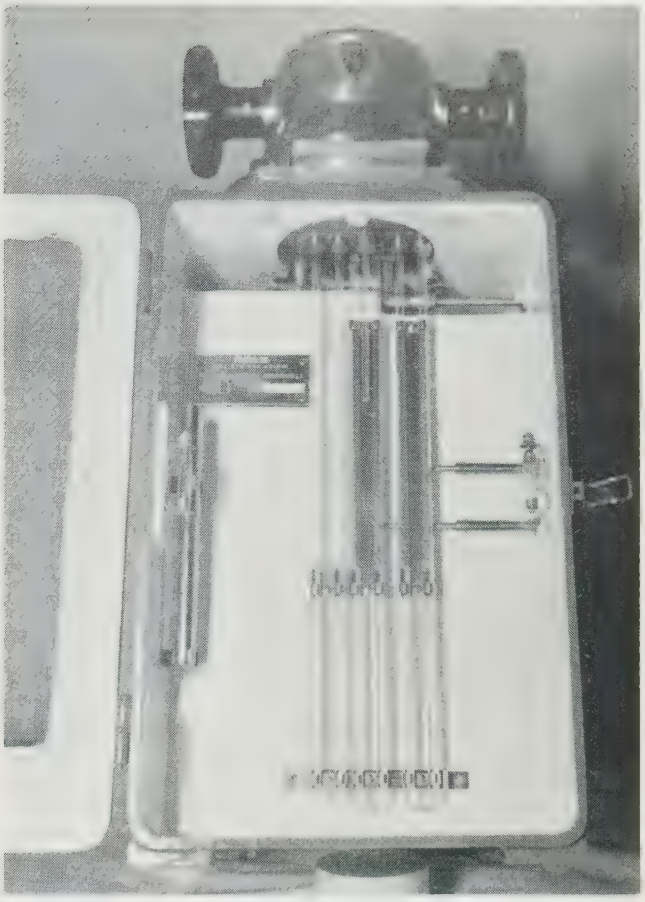

Fig. 3

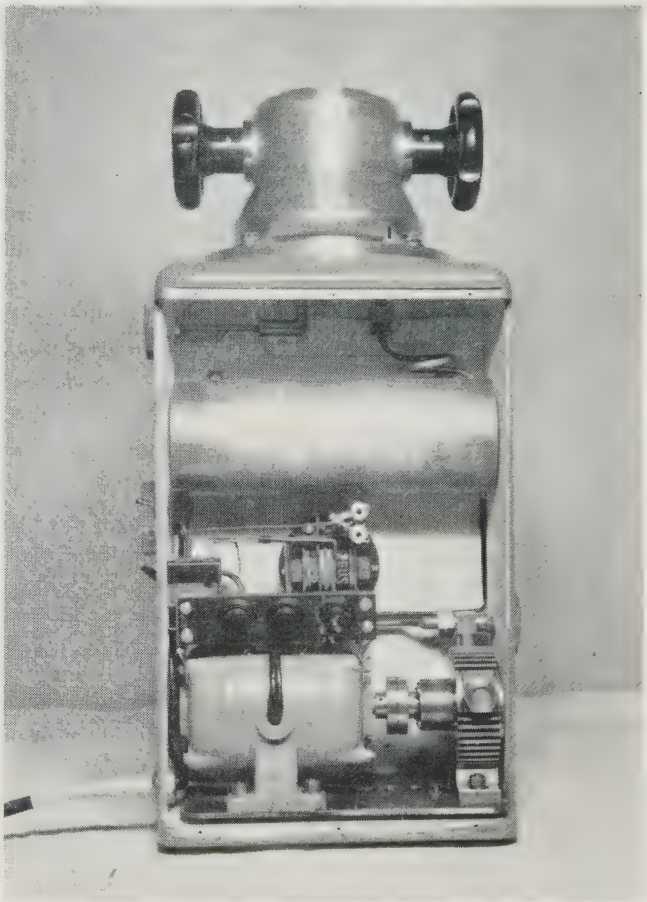

Fig. 4

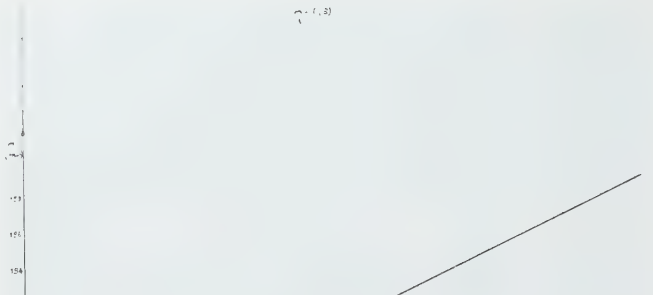

Fig. 5

EXPERIMENTAL DETERMINATION OF THE CAPILLARY CONSTANT *K*

The capillary constant is completely determined if the physical dimensions of the capillary itself are known. It is, however, very difficult to have a capillary tube with an inner diameter perfectly constant along its axis. Therefore it is preferred, in practice, to determine the constant k experimentally, using equation (2) given above. From equation (2), it follows that:

$$k = \frac{\Delta p_2}{Q\eta} \qquad (5)$$

Then if the volume flowing per time unit under the pressure difference Δp_2, and the air temperature to which a certain value of η corresponds (see Fig. 5) is determined, it is possible to calculate the value of the constant k. The apparatus used for this determination is schematically represented in Fig. 6. The principle on which it functions is as follows: From A, vessel B is filled with water, the level of which is kept constant by means of the constant level outlet C. From B, the water flows through D and fills container E which has a fixed volume between the two marks L and L′. The volume of water that enters this container corresponds to an equal volume of air that flows through the drier and the capillary H. The temperature of air is measured by the thermometer T, while the pressure difference between the input and output of the capillary is read from the water manometer M. The volume per time unit Q is simply determined by measuring the time taken for the water level to pass from the bottom mark to the top mark in container E. The enlargement in the bottom of the container ensures operating in steady conditions. The two driers F and F′ are used to prevent the entry of humidity into the capillary. In order to calculate the value of k more accurately, some correction must be made for the volume Q. The volume of air flowing from the container must be transformed into the volume that the same mass of air would have at a pressure corresponding to the average of pressures at the input and output of the capillary.

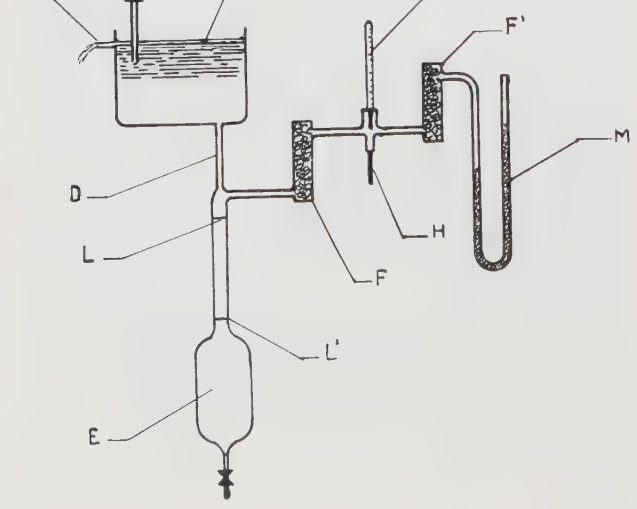

Fig. 6

Table I. Calibration of the Emanueli Tester Capillary

Number of determinations	= 50
K': mean value	= 193.512
$K'_{max.}$: max. value	= 196.451
$K'_{min.}$: min. value	= 191.229
σ	= 1.290
$\sigma\%$	= 0.66

If Δp is the pressure difference (centimeters of water) indicated by the manometer and h the barometric pressure, the volume per time unit to be introduced in formula (5), is given by:

$$Q' = Q\left(1 + \frac{\Delta p}{2h}\right)$$

Assuming that h = 1000 cm. of water; it would result that

$$Q' = Q\left(1 + \frac{\Delta p}{2000}\right)$$

and then:

$$k = \frac{980\ \Delta p}{Q\left(1 + \frac{\Delta p}{2000}\right)\eta} \qquad (6)$$

where the constant 980 is due to the transformation of water centimeters in dynes per square centimeter. As an example of the precision with which the constant k is known, Table I sets out the result of a typical calibration of the capillary. As one can see in a series of 50 determinations the per cent standard deviation is only 0.66%.

WORKING PRINCIPLE OF SCHOPPER AND GURLEY TESTERS

As is well known, in the Schopper method of determining air resistance (see schematic drawing in Fig. 7) a measured

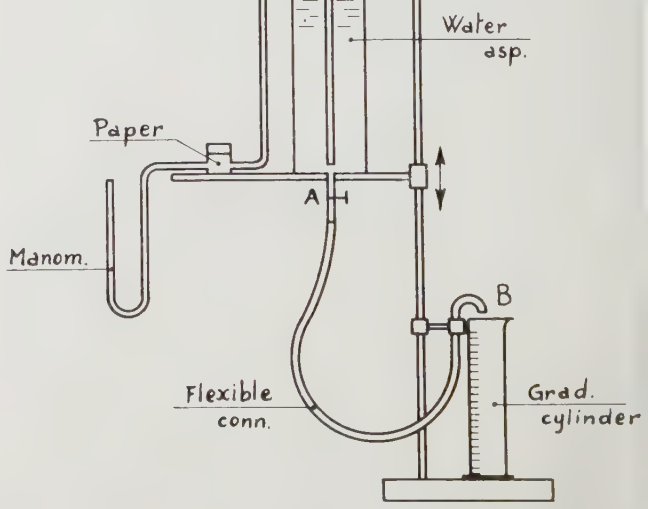

Fig. 7

volume of air is drawn through the paper being tested under the pressure difference provided by a water aspirator fitted with a Mariotte constant head device. The desired pressure difference is set out by varying the position of the point A with respect to the point B.

The Schopper porosity is given by the number of cubic centimeters of air that flows in 1 min. through a sample of paper having a surface area of 10 sq. cm. when the pressure difference is 10 cm. of water (*3*).

In the case of paper with very high impermeability, a sample with a surface area of 100 sq. cm. is taken. In the equally well-known Gurley method of determining paper air resistance there are two types of testers; the first for paper with low air resistance (standard densometer) and the second for paper with high air resistance (high pressure densometer).

The functioning principle of the two densometers is the same. The air is forced through the sample of paper under test by the sinking of an inner cylinder floating freely in an outer cylinder partly filled with light oil in the standard type and with mercury in the high pressure type. The paper is generally clamped to the base of the apparatus. Air pressure for the test is provided by the weight of the inner cylinder. The surface of the paper sample is 1 sq. in. in both types, and the pressure difference is 12.2 water centimeters in the standard densometer and 31 water centimeters in the high pressure one. The Gurley air resistance of the paper is given by the time taken by the inner cylinder to sink between marks corresponding to a fixed volume (100 cc. for the standard type and 10 cc. for the high pressure one).

In the Gurley method the air pressure difference is not constant throughout the test because the action of the inner cylinder changes continuously during its sinking. This is due to the fact that the part of the inner cylinder immersed

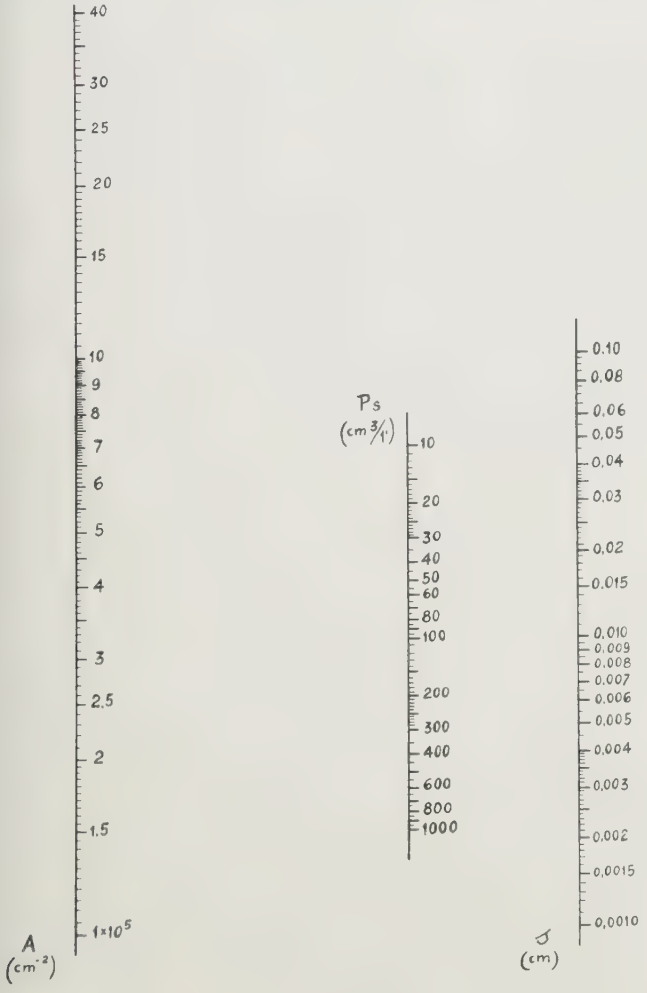

Fig. 8

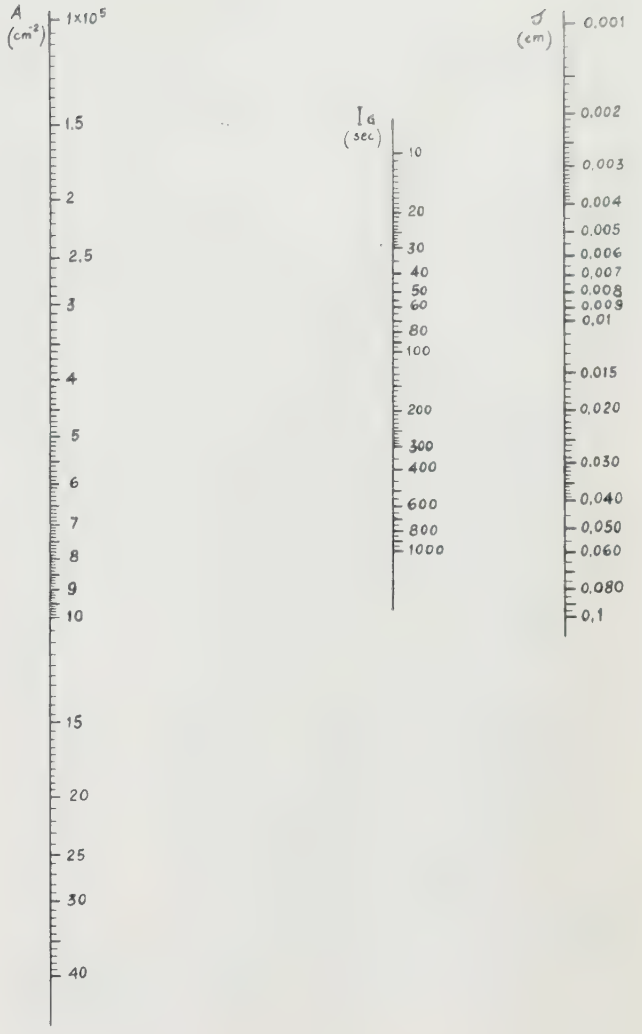

Fig. 9

in oil or mercury changes continuously, being so subjected to a continuously variable hydraulic force.

In a high pressure tester, we have found for example, that the weight of the inner cylinder, which is 167 g. at the beginning of the test, becomes 140 g. when the cylinder has sunk nearly half way and only 55 g. at the end of the test.

RELATIONSHIP BETWEEN EMANUELI AND SCHOPPER AIR RESISTANCES

The relationship between Emanueli and Schopper air resistances can be derived from the formula (I):

$$Q = 10^{-5} \frac{\Delta p_1}{\eta} \frac{S}{sA} \qquad (1)$$

In this equation, Q is the air volume per time unit flowing through the paper and is measured in cubic centimeters per second. If the Schopper air resistance is indicated by P_s, therefore:

$$Q = \frac{P_s}{60}$$

The air pressure difference Δp_1 is measured (in formula 1) in dynes per square centimeter. The pressure difference adopted in the Schopper method being 10 cm. of water, it follows that: $\Delta p_1 = 9810$ dynes/sq. cm.

Finally having $S = 10$ sq. cm. the equation (1) can be, written in the new form:

$$\frac{P_s}{60} = \frac{9810}{\eta} \frac{10^{-4}}{sA} \qquad (7)$$

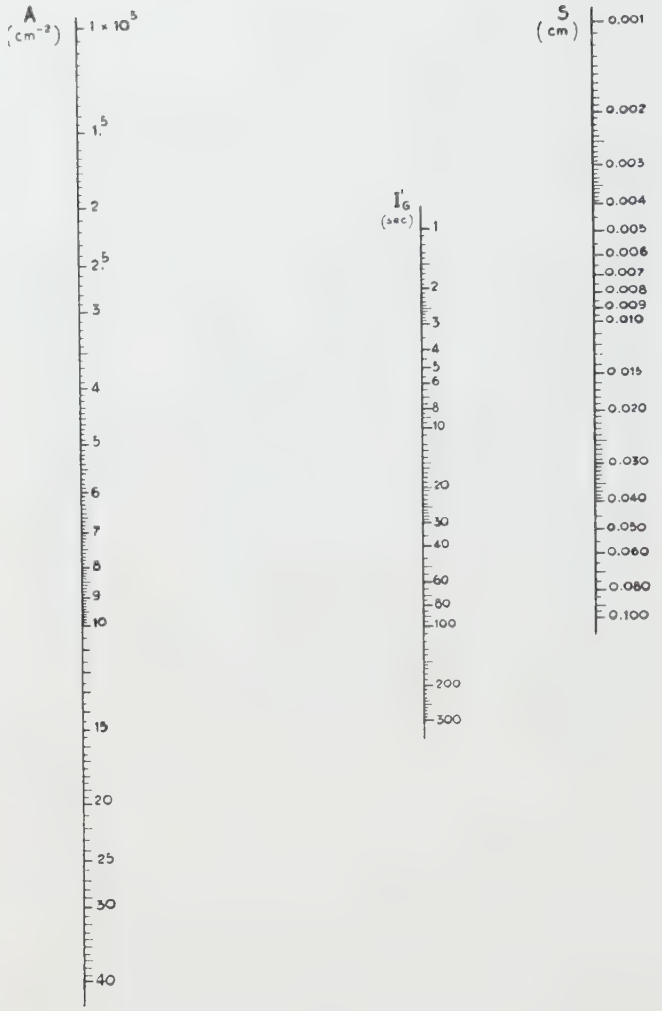

Fig. 10

If the operation is at 20°C. then the air viscosity η will have the value: 182×10^{-6} poises.

Inserting this value in (7):

$$P_s = \frac{323.4 \times 10^3}{sA} \tag{8}$$

In the case of paper having high air resistance, the surface of the sample is 100 sq. cm., therefore:

$$P_{s'} = \frac{323.4 \times 10^4}{sA} \tag{9}$$

For practical use, a nomogram (Fig. 8) has been constructed from (8) in which papers with thicknesses varying between 0.01 and 1 mm. and air resistances A from 1×10^5 to 4×10^6 are considered.

RELATIONSHIP BETWEEN EMANUELI AND GURLEY AIR RESISTANCES

The theoretic relationship established below will only be approximate because it is assumed that the air pressure difference is constant during the test. It can be, however, very useful in some cases to have a transformation formula between the two methods, even if only approximate. For establishing the relationship the equation (1) is reconsidered as:

$$Q = 10^{-5} \frac{\Delta p_1}{\eta} \frac{S}{sA} \tag{1}$$

In the case of the standard densometer, the values are as under (*5*):

Air pressure difference: $\Delta p_1 = 12{\cdot}200$ (dynes/sq. cm.)
Air volume: $V = 100$ cc.
Sample surface: $S = 6.45$ sq. cm.

If the Gurley air resistance is indicated by I_G, inserting the values given above, in (1):

$$\frac{100}{I_G} = \frac{12{\cdot}200}{\eta} \frac{6.45}{sA} 10^{-5} \tag{10}$$

If the operation is at 20°C. ($\eta = 182 \times 10^{-6}$ poises):

$$I_G = 2.3 \times 10^{-2}\, sA \tag{11}$$

For the high pressure densometer, the values are as under (*5*):

$\Delta p_1 = 30{\cdot}400$ (dynes/sq. cm.)
$V = 10$ cc.
$S = 6.45$ sq. cm.

Then, if the operation is again at 20°C, it would result that:

$$I_{G'} = 9.28 \times 10^{-4}\, sA \tag{12}$$

For practical use, nomograms derived from equations (11) and (12), respectively, are given in Figs. 9 and 10.

RELATIONSHIP BETWEEN SCHOPPER AND GURLEY AIR RESISTANCES

This relation can be obtained by a combination of equations (8), (9), (11), and (12), which can now be rewritten in a new form:

$$A = \frac{323.4 \times 10^3}{sP_s} \tag{8}$$

$$A = \frac{323.4 \times 10^4}{sP_{s'}} \tag{9}$$

$$A = \frac{I_G}{2.3_s} 10^2 \tag{11}$$

$$A = \frac{I_{G'}}{9.28_s} 10^4 \tag{12}$$

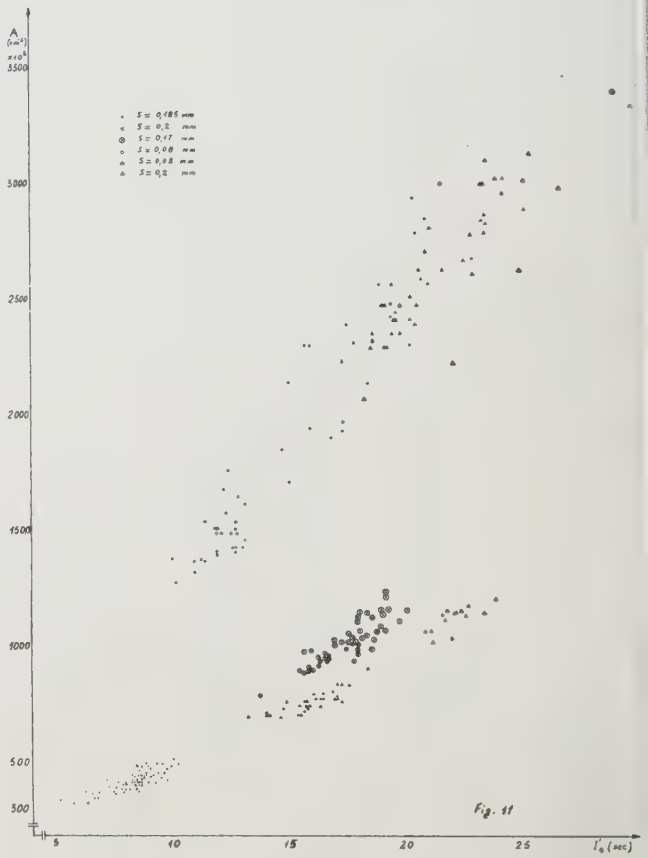

Fig. 11

Table II. Reproducibility Test

	Thickness (s), mm.	Paper air resistances expressed in Emanueli units: Mean value cm.$^{-2}$	Minimum value, cm.$^{-2}$	Maximum value, cm.$^{-2}$	σ, %	Paper air resistances expressed in Gurley units: Mean value, sec.	Minimum value, sec.	Maximum value, sec.	σ, %
Low impermeability paper	0.170	466,000	460,000	468,000	0.913	9.1	8	9.9	4.23
Medium impermeability paper	0.200	940,000	900,000	960,000	1.95	17.4	15.6	18.8	4.51
High impermeability paper	0.080	2,400,000	2,370,000	2,430,000	0.633	20.3	19.6	20.9	1.56

From (8) and (11) it would result that:

$$I_G = \frac{7.4 \times 10^3}{P_s} \qquad (13)$$

and then, from (9) and (12):

$$I_{G'} = \frac{3 \times 10^3}{P_{s'}} \qquad (14)$$

EXPERIMENTAL COMPARISON BETWEEN EMANUELI AND GURLEY METHODS

Because of the simplifications introduced in the calculation of the transformation formula between the Emanueli and Gurley air resistances, it has been thought advisable to make an experimental comparison between the two methods in order to check the formula and have an idea of the error which its use involves.

The following tests were therefore carried out:

1. Determination of the reproducibility by repeating the air resistance measurement using both methods on the same sample 50 times. The measurements were carried out on paper having low, medium, and high air resistance. For the Gurley method a high pressure densometer was used. The results of these tests are given in Table II.

2. The air resistance of about 300 samples of paper including the three above types was determined using both methods. The results of these tests are given in the graph of Fig. 11.

As can be seen from this graph the experimental values fall into different groups having different interpolating curves according to the thickness of the various samples. The thickness influence disappears when the Gurley air resistance values are made to correspond not to the Emanueli air resistances but to the product of these latter, and the thickness of the paper being tested (modified Emanueli units), as has been done in the graph shown in Fig. 12.

This graph also shows the straight line in accordance with which the Gurley values, calculated using formula (12), are plotted. From this graph it is seen that the calculated values are, as was to be expected, generally lower than the experimental values.

It may also be noted that the dispersion of the experimental values increases in proportion to the air resistance. In order

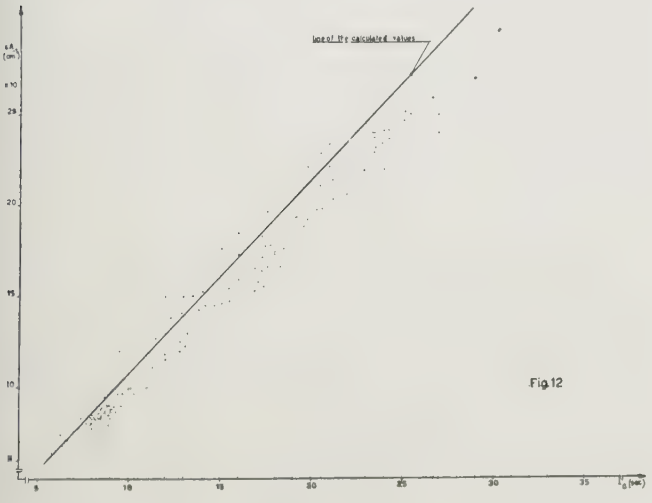

Fig. 12

Fig. 13

to give an idea of the error incurred in using the transformation formula, the experimental values being distributed asymmetrically in respect to the line of the calculated values, the average of the experimental values was compared with that of the calculated values. The global difference between the two averages, referred to the average of the experimental values, is about 5%. If the various groups (low, medium, and high air resistance) are considered, this difference varies between 4 and 7%.

SUMMARY AND CONCLUSION

The modified Emanueli air resistance tester has been described and the principle on which the Gurley and Schopper testers function has been mentioned. Some formulas were then worked out for the transformation of the three impermeability factors.

Lastly an experimental comparison was made between the Emanueli and Gurley methods and an experimental check was carried out on the formula for the transformation of the Emanueli air resistance to the Gurley one.

This experimental comparison showed that the reproducibility of the Emanueli method is decidedly superior to that of the Gurley method. The Emanueli method has also the advantage of being independent on the ambient conditions existing during the tests.

The relationship extablished between the Emanueli and

Gurley factors, bearing in mind the approximation introduced into the calculation and the errors involved in the experiments, gives reasonably satisfactory results which are at any rate, amply sufficient as a guide.

APPENDIX

Description of the first model of the Emanueli Air resistance tester: the first model of the Emanueli air resistant tester is represented by Fig. 13. In this type of tester, air is supplied by a rubber bulb (1) and passes through the paper sample which is pressed by means of the hand wheel (3) between the two flanges (2). The sample surface area is 4 sq. cm. Air issuing from the paper passes through the capillary (4) and then through (5) into the atmosphere. The pressure difference across paper and capillary, respectively, is read from the water manometers (6) and (7).

LITERATURE CITED

1. Emanueli, L., *Paper Trade J.* (Sept. 8, 1927).
2. Carson, F. T., *Bureau of Standards Journal of Research* 12: 567–585 (May, 1934).
3. Schopper, L., "Fabrik wissenschaftlicher und technischer Apparate Nr. 181," Gebrauchs-Anweisung für den Apparat zum Prüfen von Papier, Sachpapier, Filtrierpapier und dergl auf Luftdurchlässigkeit.
4. Grant, J., "A Laboratory Handbook of Pulp and Paper Manufacture," page 221, London Edward Arnold and Co. 1947.
5. American Society for Testing Materials. Designation D 726–55 T.

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Flexographic Printing Inks—Developments During the Past Ten Years and Future Prospects

RICHARD H. SCOTT

Flexographic printing has grown during the past 65 years from the crudest form of rubber stamp printing, using simple dye solutions, to its present position as the fastest growing process in the graphic arts, with its greatest progress in the packaging industry. Paper, paperboard, and especially cellophanes, gave the process its greatest impetus, with the added impact of the newer plastic films which are so readily adapted to flexographic in-line printing and finishing techniques. The improved ink formulations available today include a wide range of pigments, transparent or opaque, with better tonal values, strength, permanence, and finished product resistance. Resins, beyond original shellac, include lacquers, emulsified shellac or resin, for water dispersion systems, and the most recent protein-derived polyamide resin. Solvents, though limited by available roller and plate materials, embrace a growing range of materials, including the solvent-diluent systems utilized in the so-called "co-solvent" inks. Press utilization of flexographic inks is concerned with viscosity control and speed of drying manipulation. Both of these require knowledge of formulation and indicate close liaison with the inkmaker. The growth of the process depends on the individual and collective efforts of press manufacturer, plate and roller manufacturer, inkmaker, and papermaker, for whom much of the requirement is already defined. Better rollers, plates, stock, inks, are needed.

Flexographic printing, so long considered a minor, less important, part of the graphic arts industry, has grown in stature over the past decade, not only in the volume of its product but just as strikingly in its quality of reproduction. The concept is hardly more than 60 years old and has evolved from the crudest form of rubber stamp application to a point where it now employs some of the most imaginative design techniques available in the industry.

The earlier application of the flexographic process took the form of relatively simple marking and decorating of bags and wrapping papers. It produced its best effects when used on sharp art forms, such as large solid areas, line drawings, and bold lettering or type matter. Present flexographic printing results are limited only by those areas in press, plates, and printing inks, which have not yet developed to the degree which would produce the same quality obtainable by letterpress, offset, and more specifically, rotogravure.

In any discussion concerning flexographic printing and the present position of flexographic printing inks used in this process, we should call attention at the outset to the fact that flexographic printing has had its roots in and its greatest stimulation from the continuing growth and diversification of the packaging field. Its versatility, simplicity, and economy have accounted for its position.

Flexographic printing is now the fastest growing method of printing in the industry. It has a greater variety of application than any other single type of reproduction. Obviously, this does not pretend to answer the tremendous variety of package printing requirements. It has its limitations as well as its advantages, but there are few combination printers whom it cannot serve well in some application.

The term "flexographic" was the result of concern for the implications, in the food packaging industry, particularly, for the use of the expression "aniline" printing which heretofore had described the process. Most of this type of printing was accomplished with fluid inks, consisting of dyes dissolved in alcohol, and later, in a varnish composed generally of shellac in alcohol. The term "aniline" derived from the basic chemistry of the coal tar dye stuffs used, and came to be applied to the printing process itself.

It was in 1952, following an extensive survey of the industry carried out by the Packaging Institute, that the term "flexography" was established, and has since been used to describe the process. Flexography, as we know it, is essentially a typographic process in that the print is essentially produced from solids, type, or tone work somewhat elevated above the plane of contact. This is similar to letterpress, but, unlike letterpress, which can also take advantage of metal engravings, flexography uses rubber printing plates exclusively, either natural or synthetic. Flexography, using fluid, volatile inks, also does not require the extensive ink distribution system characteristic of letterpress paste type inks. In flexography the ink is contained in a pan or fountain in which a single roller is immersed. The fluid ink is transferred to the printing plate cylinder through a single form roller. This form roller can be rubber or, as is more characteristic in current press design, an etched steel roller. This roll, called "Anilox," permits highly improved transfer of the fluid ink to the plate cylinder. The development of this Anilox roller, like so many developments in the printing field, was the result of increasing demands for improved quality reproduction.

EARLY FLEXOGRAPHY

The early applications of flexographic printing were mainly in the field of softer paper printing and in this application the use of dye solution inks was quite satisfactory. With the advent of harder surfaced sheets, such as glassine and paperboard, and with the later development of clear cellophane, the dye-type inks, which depended in good part on absorbency for their drying, were no longer satisfactory. In addition, there had always been a problem of the transparency of the dye inks when printed on colored stocks. For example, the brown color of kraft stock completely changed or destroyed

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the tone values of the colored transparent dye. However, dyes also had other defects, principally their tendency to bleed, either one color to another, or into the stock in the presence of excess moisture. Many of the surfaces then printed would contain sufficient moisture to produce this "bleed" condition. Initial efforts to correct this problem took the form of lending opacity to these dyes by mixing them into dispersions of a pigment, such as titanium dioxide.

ADVANTAGES OF FLEXOGRAPHY

At this point it would be well to describe some of the advantages inherent in the flexographic printing process. Because of the physical properties of the rubber plates used, flexographic printing could compensate for minor irregularities in the printing surface on the softer paper stocks. Because of the ability to change plates rapidly or to insert substitute or additional plates such as those required to identify weight, price, and other special marking symbols, the cost of plating up, make ready time, and down time were considerably reduced. This made possible simple, economic, printing of short run orders. For example, an order for 5000 bags in several different identities could be run flexographic but would not be suitable to any other process.

Today, however, the emphasis has been more and more on efficiency and improved quality production at increasing output speeds. Operating costs have increased to near the level of the other methods of printing, but the lower capital investment required, coupled with the volume production possible, make flexography attractive for many applications. A considerable part of this changing pattern can be attributed to the ability of the inkmaker to meet the challenge for highly improved flexographic ink quality and performance.

IMPROVEMENTS IN FLEXOGRAPHIC PROCESS

Flexographic printing has also been the by-product of adversity. During the period of depression in the 1930's there was a considerable changeover by paper converters to the economies inherent in the flexographic process. During the war years, with printing press production at an enforced standstill, press manufacturers, with the advantage of precision engineering techniques, were able to upgrade press development to include improved web tension control, pneumatic or hydraulic pressure controls between rollers in the ink distribution system, and later, the Anilox etched application cylinder. Newer presses were designed, of heavier construction, engineered to eliminate vibration and prevent distortion of the print at higher operating speeds; with improved roll balance and better bearing characteristics.

Another engineering feature which greatly enhanced the stature of the flexographic process was the development of the "in-line" operation, that is to deliver the product from printing through some combination of waxing, slitting, sheeting, perforating, numbering, and/or die-cutting operations, to a finished product in a continuous, mechanical process.

The availability of completely pigmented ink systems, both opaque and transparent, improved the ability to produce color variety and improved printed results. The advent of transparent wrapping material, such as cellophane and the newer plastic films, proved particularly adaptable to utilization of the developments outlined above. In addition, constantly improving plate materials, and plate production techniques, permitted greatly increased application of this basically simple, economic, printing process. Each of these developments has posed concurrent problems for the other parts of the process, and present high quality results obtainable by flexography are the result of continuing improvements in all areas.

FLEXOGRAPHIC PRINTING INKS

Flexographic printing inks are now predominantly pigmented systems and we will discuss some of the features which have been, and are continuing to be, built into specific formulations. Present formulations for flexographic inks have evolved from the most primitive examples of dyestuffs dissolved in alcohol. There were later requirements for addition of a binder, usually shellac, for harder surface stocks. First attempts at pigmentation for opacity utilized dispersions of titanium white tinted with dye.

Early pigmented flexographic inks were developed to meet several distinct problems. Dye inks lacked finish, opacity, and permanence. Colors also bled in water and bled one into another when overprinted. In the open fountains, on earlier presses, alcohol would evaporate too rapidly and would also absorb water from the atmosphere, particularly on humid days. The effect on ink and print were often disastrous. In addition, with the increasing availability of cellophane in volume and variety and its obvious adaptability to flexographic printing, lack of opacity and resistance to bleeding in film plasticizers had to be overcome.

With the increasing ability of flexographic press equipment to handle all the available types of paper, board, cellophane, and later, transparent plastic films, the earlier simple flexographic ink formulations have been displaced by materials as sophisticated as any others produced for the other printing processes. These formulations became available as the demands for color strength and permanence, finish, adhesion, scuff resistance, and other properties were dictated for quality reproduction equal to that which could be achieved by other methods.

Flexographic inks are composed of pigment, or pigment-dye combinations, resin, or combinations of resins and other film formers, solvents or mixtures of solvents, with or without diluent. Formulations in specific cases will also contain materials for improved properties with respect to block resistance, scuff resistance, or other product requirement properties.

Pigments

The pigments used in these inks are the standard whites, blacks, as well as a full range of inorganic and organic colors. The specific type of colors will be dictated by particular types of ink as well as by special requirements for permanence or end-product resistance. For example, pigments used in the formulation of water dispersion systems have had to be selected, or in some cases specifically developed, for proper functioning of the ink at the press. Certain so-called resinated pigments have produced poor flow characteristics and extremely limited body stability. Another requirement has been the ability of specific colors to resist chemical reaction, either with other ingredients, or with particular types of coatings or particular products to be packaged. Certain pigments are not wax resistant, a requirement for milk carton printing: other pigments are not resistant to organic fats or fatty acids. The entire range of available organic pigments has had to be carefully evaluated to determine the particular pigments suited for a given type of printing. In fact, completely new lines of pigments have become available over the last decade as a result of the inability of existing colors to meet specific requirements.

Resins

The resins used in flexographic inks have also gone through a period of improvement and adaptation. The resins available subsequent to the utilization of shellac in early formulations had to be soluble in the rather limited numbers of solvents which can be used in this rubber plate process. Current formulations employ various grades of nitrocellulose lacquers and many synthetic resins developed for alcohol solubility, as well as, improved finish, nonblocking tendencies through better solvent release characteristics, heat resistance, and other properties. The most striking addition to the list of available materials has been the protein-derived polyamide

resins which have increased the versatility of flexographic ink systems tremendously.

Inks which were used for printing earlier grades of polyethylene films usually employed shellac in the resin binder. These inks exhibited poor water resistance properties, a feature required in the expanding wet-pack vegetable field. At first, attempts were made to correct this problem by overprinting, but this produced higher costs which ruled out its use.

The availability of the protein-type resins and, more important, the increasing ability of printing ink technicians to work with these materials, fitted the requirement for water resistance, and opened up a new approach to many of the other specific requirements of the tremendous packaging field for the flexographic process. Printing inks, based on these materials, produced better block resistance, increased gloss, excellent adhesion, and water resistance.

The development of these improved inks, however, posed new problems for the printer, in that these co-solvent inks, as they are commonly called, used a combination alcohol solvent and aliphatic diluent. Ordinary rubber press rollers and plates could not carry these materials without swelling and distortion. Synthetic rollers and plates had to be used for effective results and printers were reluctant to switch from existing rollers for specific jobs. Different solvents and mixes were required for press reduction of these inks which also complicated inventory and press adjustment techniques. Much of this has been overcome as more and more printers have turned to synthetic rubber with the attendant improvements in these materials by roller and plate manufacturers. Flexographers have learned to handle the different solvents involved and inkmakers have developed newer formulas which minimize the problems of press adjustment.

Another important development in flexographic inks came about when inkmakers produced alkaline water dispersions of shellac and other resins. While used initially on cheaper paper printing, it has found tremendous application in the milk container industry, and in the printing of multiwall bags and other kraft papers. It has displaced a major portion of this printing, done formerly with moisture-set inks, for lower initial investment, printing speeds possible, fast drying, and lack of offsetting.

Solvents

Solvent usage in flexographic ink formulation has been limited by the very nature of the process. Unlike rotogravure, where almost an unlimited range of solvent types may be employed, flexography has been limited by the characteristics of rollers and plate materials to a much more restricted number. The most widely used alcohol is proprietary grade ethyl alcohol. To control drying rates methyl alcohol may be used to speed up drying, but should only be used to a very limited extent because of its flammability and possible toxic effects. Excessive evaporation rates may also condense moisture into the ink causing the resin to become insoluble. To slow the speed of drying, small amounts of Cellosolve may be used. Control or manipulation of drying speeds must be handled with care. An ink which is too slow drying may be more difficult to control for uniform print quality, and, if inadequate drying is obtained through the press equipment, serious blocking problems may result from trapped solvent resoftening the ink film. An ink which is too fast drying may dry in on rollers and plates affecting distribution and printability. Type matter and light areas on the print will fill in, while the edges of the plate will begin to scum. The technical assistance of the inkmaker, who has knowledge of the original formula solvent balance, will avoid the occurrence of these problems.

Water may also be considered a solvent for those flexographic inks which are essentially alkaline, water emulsion type. Water is more a diluent here than a true solvent. Drying is accomplished in large part by absorption into the porous stocks employed and also by evaporation. While this type of ink is not too far from the costs of other types, the economies obtainable result from the use of the cheaper, more absorbent, paper surfaces, the ability to use water for press reduction, and the minimizing of fire hazard. In certain cases, it is possible to adjust drying speed by utilizing small amounts of the alcohols and other solvents mentioned above.

With the protein-derived resin systems we have referred to earlier, combination solvent-diluent systems are employed. Usually a combination of an alcohol with a low solvency aliphatic hydrocarbon is chosen for high resin concentration and good body stability. Specifically, for faster drying, isopropyl alcohol with lactol spirits, or toluol, should be used, and, for slower drying, normal propyl alcohol should be used with VMP naphtha. It is also important that any additional solvent required for these inks, either to make them press-ready, or to maintain viscosity or color control at the press, should be premixed in the ratio suggested by the inkmaker and then added carefully to the ink with sufficient mixing. Some types of these newer ink systems can be thinned with just isopropyl or normal propyl alcohol. But care must be exercised to avoid indiscriminate mixing of these apparently similar inks. Since they are based on a solvent, or solvent combination, with limited solvency power, the original formula balance must be maintained through any mixing or solvent addition.

There are some special low cost printing applications on inexpensive absorbent stock where a system based on a resin soluble in aliphatic hydrocarbon may be used. Here a solvent such as VMP naphtha or lactol spirits with lower solvency power and limited boiling range, may be used. These applications require synthetic rubber on the press for best work. When the newer plastic films, such as pliofilm or vinyl, are run by flexography, even stronger solvents will be required, and even synthetic rollers and plates may not be sufficiently resistant to the dissolving or swelling action of these solvents.

Additives

In addition to the three prime ingredients, pigments, resins or binders, and solvents, which have been described, there are a number of additive materials, resins, waxes, oils, used either alone or made into compounds, which will give special performance properties to the flexographic ink formula. Such things as improved smoothness and leveling of print, better rub resistance, printed film flexibility, and other characteristics can be effected by the proper addition of small percentages of such materials.

PRESS APPLICATION

All flexographic ink formulations, whether the simple or more complex type, are only as effective as proper stock and press operating techniques can deliver. There are two basic operating characteristics on which flexographic inks depend for quality, uniform printed results. This assumes, of course, that laboratory formulating and testing have demonstrated that the particular ink can deliver the properties required. These operating characteristics are viscosity and speed of drying. Viscosity, properly controlled, will insure that, when other press conditions are maintained, a uniform film of ink will be deposited on the surface being printed. Unlike rotogravure, which can closely predict the amount of ink deposited by the depth of cylinder etch, flexography depends entirely on adjustment of the contact between rubber rollers and rubber printing plates to give proper print tonal values. This impression must be a "kiss" contact, that is, to produce the minimum film thickness, at the proper viscosity, which will give the color tonal values required. Once obtained, this balance will depend on proper viscosity control to maintain uniform results. This viscosity

control can be manual, that is, by checking with a hand unit, such as a Zahn cup, at frequent intervals, or it may be automatic, sampling the fountain ink continuously and, by automatic valve manipulation, adding the proper amount of solvent to maintain viscosity. In either case, solvents or ink additions should be made carefully. The additions should be mixed into a reservoir to the ink fountain by means of a circulating pump to avoid sharp erratic changes in printed result.

Speed of drying will depend not only on the film thickness deposited but on the speed of the press, and on the size and caliber of drying equipment on the press. The characteristics of the stock will also affect drying. On softer, absorbent papers some drying will result by absorption into the pores of the stock. On harder surfaced stock most of the drying will have to be by evaporation. Generally speaking, flexographic inks should have a balanced solvent system. But it should be remembered that it is much easier to carefully slow down drying by proper additions, than it is to speed up drying if a percentage of slow solvent is already in the ink. Not only will drying speed be retarded but blocking and film resoftening may result. Solvent additions should not be made directly to fountain, but should be premixed, either separately, or in a reservoir, so as not to disrupt formula balance or print characteristics.

POTENTIAL UTILIZATION OF FLEXOGRAPHY

Flexography has found a sure and growing place in the graphic arts, particularly in the packaging field. Its versatility, simplicity, and economy have made it the fastest growing process in the package printing industry. Most of the development work in presses, rollers, plates, and inks has been to serve this requirement. But the application of the process is increasing in many other areas including envelopes, business forms, and even in publication work. When the printing industry recognizes the possibilities in flexography, demands for technical improvement will be placed on the paper industry for smoother, more uniform printing surface capable of producing and maintaining gloss, on the plate and roller industry for solvent resistant materials to permit printing or any and all types of surface or film without swelling or distortion, and for a material capable of accepting tone work, comparable to other processes, and having the resiliency and durability necessary to produce long enough printing runs without a breakdown in the quality of tone work because of a deterioration or fatigue failure in the base material. Inkmakers need to do some very basic work on the problem of improved transfer of ink film from roller to plate to print. This will have to involve plate and roller manufacturers sooner or later, but the ability to better control ink receptivity on plate or paper, to predict film splitting characteristics, so as to produce optimum and uniform film transfer throughout the system, eventually will give flexographers the ability to more and more fully utilize the capabilities of their process. Flexographic inks need improvements in gloss for film surface printing, better heat seal properties, and development for laminating techniques and boilability, these latter requirements coming from current demands in the packaging and frozen food fields. There is a need for improved wet-rub resistance in flexographic ink systems with the growth of polyethylene coated paperboard in the milk carton field. Some of the newer polymer coated cellophanes need improved formulations for finish, adhesion, aging characteristics, and other properties. Flexography too has had a touch of the "universal" ink, with its tempting economies in press operation, inventory, and solvent utilization. Perhaps, there is an area, if some stabilization occurs in this rapidly growing industry, in which formulations can be developed which will be adapted to many surfaces and which will require a very limited number of solvents, for press manipulation. These changes will occur, as is characteristic of the printing industry generally, when developments in one phase of its activity force improvements in other areas.

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Organo-Phosphorus Epoxides

LEE M. KINDLEY, LOUIS P. GLEKAS, and PAUL E. RITT

Two general methods were explored in an attempt to prepare organo-phosphorus epoxides capable of being cured to thermally stable epoxide resins. These were: (1) the condensation of epichlorohydrin with organo-phosphorus compounds having two or more -OH groups, and (2) epoxidation of unsaturated tertiary phosphine oxides. The preparation of a phosphorus-containing epoxide resin by condensing tris(hydroxymethyl)phosphine with epichlorohydrin was investigated in some detail. A route for preparing a phenolic tertiary phosphine oxide is described. The preparation of unsaturated tertiary phosphine oxides and subsequent epoxidation with a peracid to yield a phosphorus epoxide is described. The experimental work described represents only the initial research stage in the investigation of a novel idea—that of improving the thermal stability of an epoxide resin by incorporation of phosphorus in the molecule.

Epoxy resins have enjoyed one of the fastest developmental growth rates of the many plastic materials now used in commercial applications. Even more impressive than this rapid growth rate is the variety of applications to which epoxy resins have been put in the short time since they reached commercial importance. Due to their outstanding strength, adhesion, and chemical resistance, these resins have played an important role in broadening the total utility of plastics in industry. Epoxy resins are widely used today in dielectric coatings, in resins for encapsulating electronic components, and in fiberglass laminates for printed circuits. These represent a few of the applications of plastic materials which are of vital interest to Melpar and the electronics industry.

Increasing requirements for thermally stable polymers suitable for such applications have prompted research on the modification of epoxy resins. There is considerable evidence to show that certain semiorganic polymers have unusual thermal stability. In particular, certain organophosphorus compounds have received considerable attention as possible candidates for making high-temperature polymers. The high stability of P—C and P=O bonds seemed to justify a program directed toward modification of epoxide resins by incorporation of phosphorus as an integral part of the epoxide molecule.

Melpar undertook this program, which was supported in part by the U. S. Air Force under a contract monitored by The Materials Laboratory of Wright Air Development Division.

The initial phase of the work included an evaluation of the thermal properties of a number of commercial epoxide resins by thermal gravimetric analysis, differential thermal analysis, and heat of distortion measurements. The second phase of the program involved the actual synthesis and polymerization of organophosphorus epoxides. In the final phase, which is still to be completed, the thermal data obtained for the commercially available epoxide resins will be compared to that shown for phosphorus epoxide polymers.

THERMAL PROPERTIES OF COMMERCIAL EPOXY RESINS

The determination of the thermal properties of a material is subject to wide variations. A comparison of test results

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is apt to be misleading unless the test procedure is clearly defined. For this reason representative commercial epoxy resins were subjected to three different thermal stability tests.

The heat of distortion temperature test closely followed ASTM Designation D 648–45 T. Figure 1 shows a picture of the apparatus. The test consisted primarily of supporting the sample, applying a load, and observing the temperature at which deformation occurred.

Differential thermal analysis (DTA) was used to determine thermal stability by indicating the magnitude and duration of endothermic or exothermic reaction as a function of sample temperature. Figure 2 shows the arrangement of the equipment. Furnace temperature is increased at a rate of 5°C. per min. by a programmed increase of power, and the change of temperature is plotted as a function of temperature on an X-Y recorder. The first prominent peak in the DTA curve was taken as the "end point."

Thermal gravimetric analysis was used to determine composition changes during the heating of a sample. A schematic diagram of the apparatus is shown in Fig. 3. Continuous weighing of the sample at elevated temperature is necessary. In operation, the furnace temperature is elevated at roughly 5°C. per minute by a controlled variac setting. If the sample loses weight, a slug will rise inside the transformer and produce an increased output. If the sample increases in weight, the slug lowers toward the center of the transformer producing, at first, a lower output until the "null" is reached, then a greater output. Weight change is plotted as a function of temperature.

Table I shows the thermal stability of the samples as interpreted from all three methods of determination.

The results from differential thermal analysis are somewhat difficult to explain, but thermal gravimetric analysis curves show a well defined break in the temperature range where disruptive volatilization first becomes rapid, thus enabling clearer interpretation of degradation temperature.

SYNTHESIS AND POLYMERIZATION OF ORGANOPHOSPHORUS EPOXIDES

The second phase of the program involved the synthesis

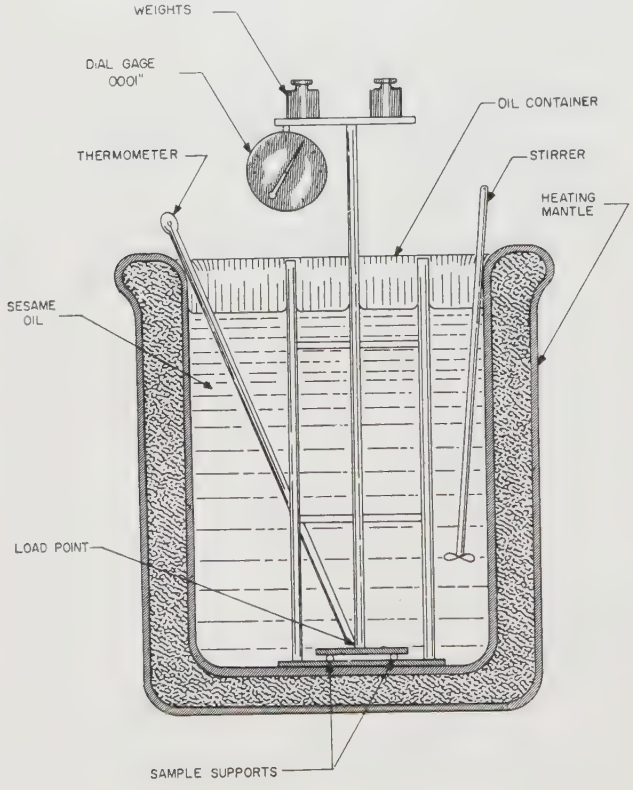


Fig. 1. Heat of distortion apparatus

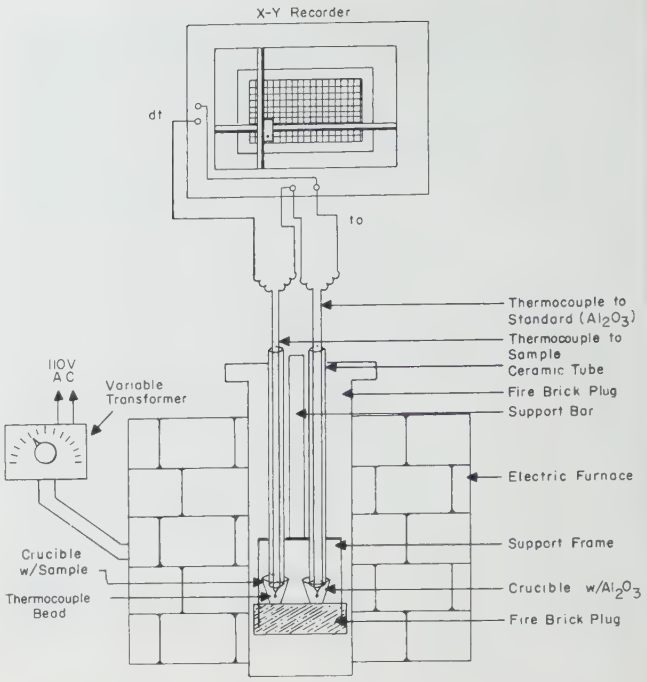


Fig. 2. Arrangement of DTA apparatus

and polymerization of organophosphorus epoxides. This work is currently in progress. The preparations follow two main routes: (1) condensation of epichlorohydrin with organophosphorus compounds having two or more OH groups; and (2) epoxidation of the double bonds of unsaturated tertiary phosphine oxides. The organophosphorus epoxides obtained will be cured by the usual amine and anhydride curing agents.

Tetrakis(hydroxymethyl)phosphonium chloride (THPC) was selected as a multifunctional phosphorus compound for reaction with epichlorohydrin. This compound is the salt of a weak base and a strong acid, and has a pH in aqueous solution of approximately 2. In the presence of alkali, THPC liberates formaldehyde and hydrogen to form tris-(hydroxymethyl)phosphine oxide (THPO).

$$\begin{matrix} P(CH_2OH)_4 \\ | \\ Cl \end{matrix} + NaOH \rightarrow \begin{matrix} P(CH_2OH)_3 \\ \| \\ O \end{matrix} + CH_2O + H_2 + NaCl$$

It has been reported (*1*) that THPC is converted to THPO during reaction in alkaline, neutral, or mildly acidic solutions, or when heated. Under these conditions the compound acts as a trifunctional monomer.

Initially the reactions of epichlorohydrin and THPC were carried out in the presence of a slight excess of base as is employed in the commercial epichlorohydrin/bis-phenol reaction. In these reactions it was postulated that the hydroxyl units of the THPC would behave similarly to the phenolic hydroxyl in the typical epichlorohydrin/bis-phenol reaction. The base would act as a catalyst and as a neu-

Table I. Thermal Stability of Commercial Epoxides

Sample	*Heat distortion, °C.*	*DTA, °C.*	*TGA, °C.*
Eccomold L 266	350	340	375
Stycast 2662	..	355	350
Maraset 617	..	325	350
Sealcast 506	325	320	312–325
Epon 828/PMDA-MA	..	313	317
Epon 828–Epon × 1310	..	..	..
1	295	296	350
2	298	296	365
3	303	269	350
Epoxylite 813	290	330	325
Dow Epoxy Novalac × 2638.3	254	340	330
Permacel ST 3994	199	293	..

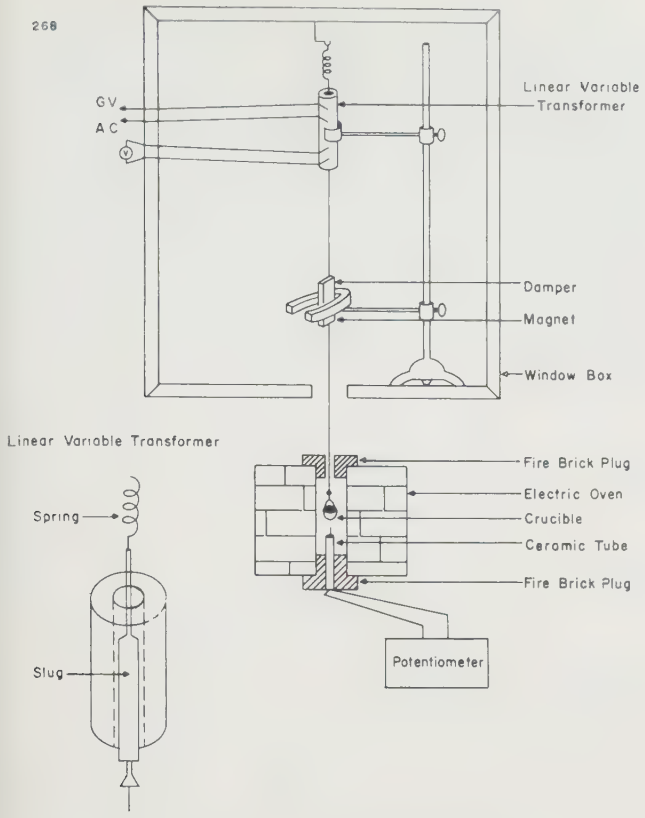


Fig. 3. Arrangement of TGA apparatus

tralizer for the hydrochloric acid which is formed. Various mole ratios of epichlorohydrin and THPC were reacted, varying the time and temperature of the reaction and amount of sodium hydroxide solution.

The products from each of these reactions were water soluble. The infrared spectra of these products showed very intense hydroxyl absorption bands with only slight indication of the presence of epoxy groups.

Amines and acid anhydrides were used as curing agents for these products. Curing with amine type curing agents such as BF 3-400, a boron trifluoride amine complex, gave water-soluble foam products. Anhydride type curing agents such as pyromellitic anhydride and maleic anhydride gave hard, water-insoluble foams.

In previous runs in which aqueous sodium hydroxide was used to condense THPC and epichlorohydrin, there was evidence of considerable hydrolysis and little epoxide formation. It was thought, therefore, that the THPC hydroxyls might react more directly as alcoholic hydroxyls. Although aqueous sodium hydroxide can be employed successfully to produce glycidyl ethers by the condensation of phenolic hydroxyls with epichlorohydrin, the condensation of alcoholic hydroxyls and epichlorohydrin to give glycidyl ethers is usually carried out in two steps. The first step is an acid catalyzed condensation carried out under anhydrous conditions to give the chlorohydrin. The second step is a dehydrohalogenation with a base to give the epoxide.

If it can be assumed that the hydroxyl groups of THPC behave as alcoholic hydroxyls, an acid catalyzed reaction with epichlorohydrin might be expected to proceed as follows:

$$(HOCH_2)_3P(Cl)—CH_2OH + CH_2(O)CH—CH_2Cl \xrightarrow{\text{Acid}} (HOCH_2)_3P(Cl)—CH_2—O—CH_2—CH(OH)—CH_2Cl$$

The reaction is not limited to the condensation of one mole of epichlorohydrin with a mole of THPC as shown above. Theoretically each of the hydroxyl groups of the THPC is capable of reacting with a mole of epichlorohydrin. The subsequent treatment of the chlorohydrin with strong NaOH should effect ring closure to give the epoxide. Since it is known that THPC in aqueous base is converted to THPO, it was postulated that the second step of the reaction would proceed as follows:

$$(HOCH_2)_3P(Cl)—CH_2—O—CH_2—CH(OH)—CH_2Cl + NaOH \rightarrow (HOCH_2)_2P(O)—CH_2—O—CH_2—CH(O)CH_2 + CH_2O + NaCl + H_2$$

The instability of THPC raises certain questions concerning the probable course of the reaction. Decomposition to THPO may occur during the initial acid catalyzed reaction, in which case only three hydroxyl groups would react and the product after reaction with three moles of epichlorohydrin would be:

$$P(=O)(-CH_2OCH_2—CH(OH)—CH_2Cl)_3$$

On the other hand, it is quite possible that under mild anhydrous conditions, all four hydroxyls would react. In this case, decomposition to the tertiary phosphine oxide would not occur even during the ring closing step with base, and the final product would have the structure:

$$P(Cl)(-CH_2O—CH_2—CH(O)CH_2)_4$$

In conducting this reaction it was found that both steps were highly exothermic and difficult to control. A method was developed for controlling the reaction in the first step, but in the second step there was either a violent exothermic reaction resulting in a mixture of crosslinked, insoluble polymeric products or a product which contained no detectable epoxide and failed to cure with the usual epoxide curing agents.

It has been pointed out that THPC is converted to THPO during reaction under alkaline, neutral, or mildly acid conditions or when heated. During the course of our work, purified THPO became available from the Hooker Chemical Corp. By using this compound as a starting material, it was reasoned that some of the complications caused by the instability of THPC could be eliminated, and a clearer picture of the expected reactions could be realized. The reactions proposed for the two-step preparation of the epoxide from THPO and epichlorohydrin are essentially the same as the reactions proposed for THPC and epichlorohydrin mentioned previously. However, the reaction in which THPC decomposes to THPO is eliminated since THPO is the starting material.

The first step of the reaction is represented by the acid catalyzed condensation of THPO and epichlorohydrin under anhydrous conditions.

$$P(=O)(-CH_2OH)_3 + 3CH_2(O)CH—CH_2Cl \xrightarrow{\text{Acid}} P(=O)(-CH_2—O—CH_2—CH(OH)—CH_2Cl)_3$$

Subsequent treatment of the chlorohydrin with a base should effect ring closure to give the epoxide.

$$P(=O)(-CH_2—O—CH_2—CH(OH)—CH_2Cl)_3 + \text{base} \rightarrow P(=O)(-CH_2—O—CH_2—CH(O)CH_2)_3 + Cl^- + H_2O$$

Table II

Sample	Curing agent	Curing conditions	Description of cured product
1	Phthalic anhydride 30 PHR[a]	24 hr. at 120°C. 2 hr. at 200°C.	Thermoplastic Solid at room temperature Softens at about 80–100°C.
2	Pyromellitic anhydride 55 PHR[a]	20 hr. at 220°C.	Rigid nonuniform foam
3	Curing agent Z[b] 20 PHR[a]	2 hr. at 80°C. 2 hr. at 150°C.	Rigid nonuniform foam

[a] Parts per hundred resin.
[b] Shell Chemical Co.

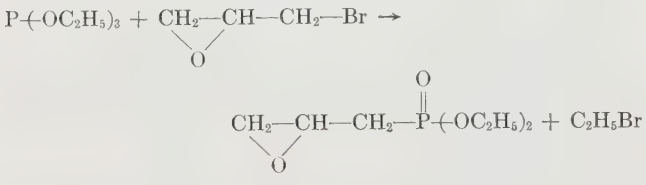
Investigation of these reactions is not yet complete, but a considerable amount of progress has been made. It appears that the acid catalyzed condensation proposed for the first step does occur. This conclusion is based on the fact that a completely homogeneous liquid is obtained which contains practically no oxirane oxygen. Because of the difficulty in obtaining a pure sample of the reaction products, positive identification and structure proof have remained a problem.

Ring closing experiments have been attempted with a number of the most promising condensation products. These experiments indicated that some ring closure took place since the percentage of chlorine decreased significantly. The amount of chlorine remaining, however, indicated that the reaction was far from complete. This was confirmed by the fact that the amount of oxirane oxygen was less than the amount calculated for the proposed reaction.

In polymer chemistry, it is often found that useful products can be obtained from reactions which are not as well characterized nor as complete as desired. Therefore, some of the samples obtained from the THPO-epichlorohydrin were cured with various epoxy curing agents. The results of the most promising samples are summarized in Table II.

In addition to THPC and THPO, other organophosphorus compounds were considered in the program for developing heat stable epoxy resins. The preparation of other phosphorus compounds containing one, two, and three epoxide groups is currently being investigated. It is proposed that these compounds be self-polymerized or used as reactive modifiers with commercial epoxide resins.

The compound, diethyl 2,3-epoxypropane phosphonate, described previously in a patent (*2*), was prepared in our laboratory by reacting triethyl phosphite with epibromohydrin as follows:



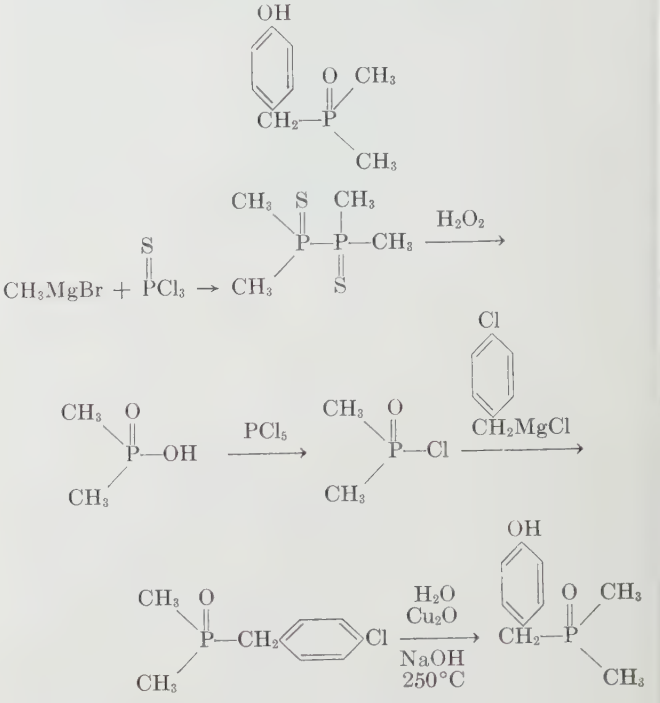
It was thought that this compound might prove useful as a reactive resin modifier. Diethyl 2,3-epoxypropane phosphonate is completely compatible with such commercial epoxide resins as Epon 828.* It greatly reduces the viscosity of the commercial product which is often advantageous in casting and laminating applications. During cure, the modifier becomes an integral part of the polymerized product due to the reactivity of the epoxide group. It does not tend to evaporate or "bleed" during or after curing but remains as a permanent plasticizer in the resin.

The synthesis of phenolic tertiary phosphine oxides such as

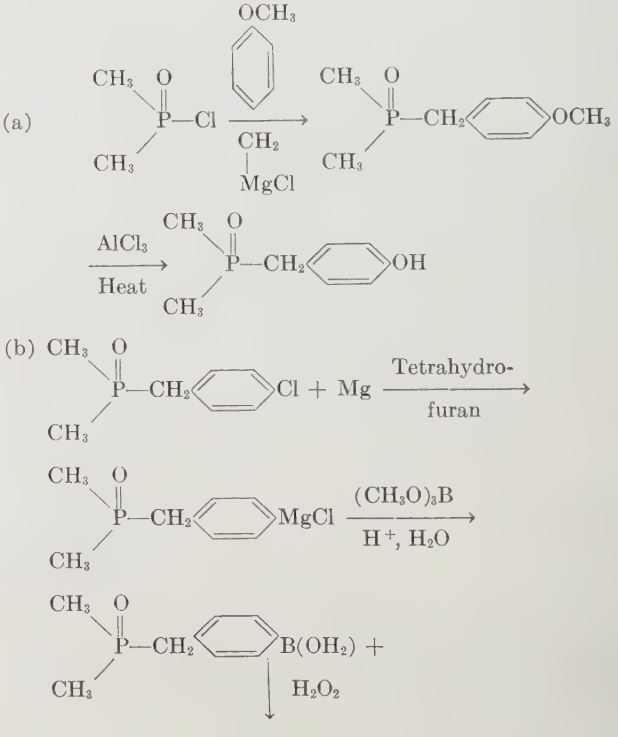
* Shell Chemical Co.

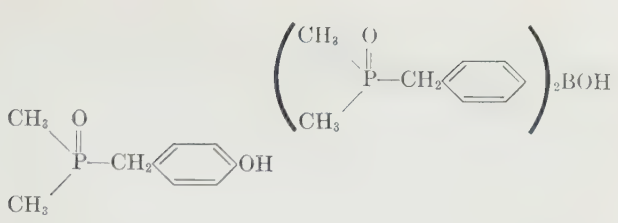
has been undertaken in our laboratory. Such compounds should react with epichlorohydrin in the presence of a base to give the epoxide in a manner similar to the reaction of epichlorohydrin and bis-phenol. To test the effect of functionality, mono-, di-, and trifunctional tertiary phosphine oxides are being prepared. Since very few of the intermediates necessary for the preparation of these phenolic tertiary phosphine oxides are available commercially, we have initiated a rather extensive synthesis program for obtaining these compounds. The synthesis routes for the mono-, di-, and trifunctional compounds may be summarized as follows:

1. Monofunctional compound: *p*-hydroxybenzyldimethylphosphine oxide

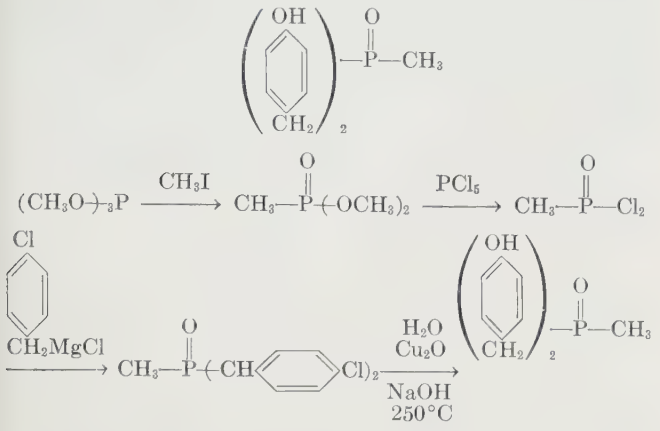


Preliminary experiments on two alternate methods for synthesis of the above phenolic tertiary phosphine oxide are in progress. These are



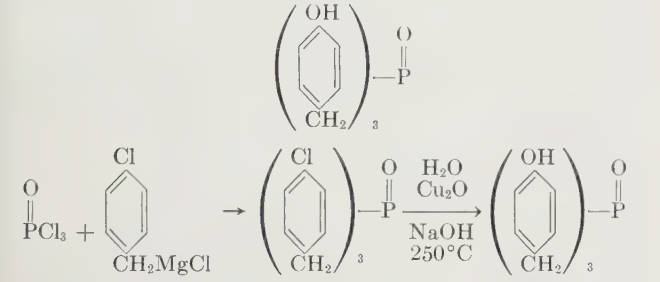


2. Difunctional compound: bis(*p*-hydroxybenzyl)methylphosphine oxide



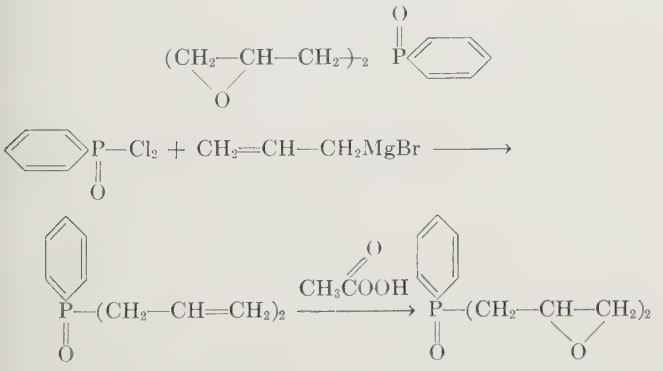
The above compound has been prepared and reacted with epichlorohydrin to yield a resinous product. Characterization and testing of this product are planned for the immediate future.

3. Trifunctional compound: tris(*p*-hydroxybenzyl)phosphine oxide



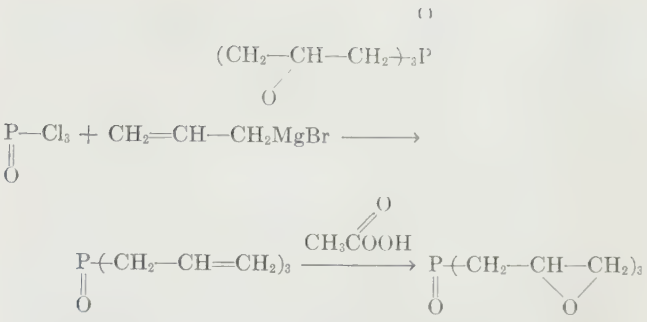
Another approach initiated in our laboratory is the preparation of unsaturated tertiary phosphine oxides and epoxidation of these compounds. The synthesis routes planned for making di- and trifunctional epoxides may be summarized as follows:

1. Difunctional compound: diglycidylphenylphosphine oxide



The unsaturated compound has been prepared and polymerized by free radical initiators. Both perbenzoic and peracetic acid have been used for the epoxidation. The final product obtained with both peracids appeared to contain but one epoxide group. Further investigation of the reaction is currently being pursued.

2. Trifunctional compound: triglycidylphosphine oxide



It should be emphasized that all of the end products and many of the intermediates in the preparations described are new organophosphorus compounds. Therefore, a considerable amount of development work is necessary before these products can be prepared in sufficient quantity to polymerize and test as organophosphorus epoxide polymers. The work described represents only the initial research phase of a novel idea—that of improving the thermal stability of an epoxide resin by incorporation of phosphorus in the molecule. The feasibility of the idea will be proved or disproved by future developmental and test data.

LITERATURE CITED

1. Reeves, W. A. and Guthrie, J. D., *Ind. & Eng. Chem.* 48 No. 1: 64–7 (1956).
2. U. S. pat. 2,770,610.

The authors gratefully acknowledge the help of Dr. G. M. Kosolapoff whose suggestions assisted the report of laboratory synthesis. Similarly, the authors are indebted to Dr. J. L. Pentecost for substantial assistance in the work on thermal measurements.

Design of Experiments

DORIAN SHAININ

This exposition of the fundamentals of statistically designed experiments and their analysis emphasizes the problems encountered in pulp and paper as experimenters want to include many factors in their tests. A process-of-elimination method for homing in at first on the controlling factors is illustrated by a synthetic case study. The appropriate statistical design employed is a random sample of a full-factorial population.

Making improvements in processes and products can be a challenging and personally gratifying series of experiences. But understandably, in these cases, the accompanying experiment should not drag on for years to solve a single problem.

Too many managements in the pulp and paper industry sincerely feel they are beset with a series of endless and ever-changing variables. After all, is it unusual to have had numerous hypotheses advanced about the causes of a problem, conducted tests with each, and found them wanting?

Several of the readers have heard of statistically designed experiments serving as a better way of conducting tests. More than one factor at a time can be tested. Such planned experiments also permit analyses of results as caused by each factor separately (main effects) and beyond that by specific combinations of factors (interactions).

The purpose of this paper is to give a layman's version of the principles of statistically designed experiments and of their analysis, known as ANOVA (analysis of variance). But of infinitely more importance is the method described here for "discovering" the factors or variables to be so tested.

CONDUCTING AND ANALYZING AN EXPERIMENT

First, an explanation might be in order as to what is meant

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by "statistically significant results." Suppose the effect of consistency in the headbox upon basis weight is being evaluated.

	C_L	C_M	C_H
	31.8	32.5	32.8
	32.1	32.3	32.5
	32.0	31.9	33.0
	30.9	32.6	31.8
Averages	31.7	32.3	32.5

The above, single-factor results (consistency only being changed in production) represent four basis-weight determinations when the headbox consistency was low (C_L); four more when it was medium (C_M); and four when it was high (C_H).

Looking across the first row of data we see basis weight increasing with consistency; as also happens across the second row. But in the third row the result under C_L or C_M is a bit out of line; as is that under C_M or C_H in the fourth row. Thus the advantage of considering a rather well-known statistical principle seems clear here: averages will tend to be closer to "true" values than are individual results. So the three average values are better estimates of the effect of consistency on basis weight.

What are the chances that this effect is real; that it can be expected to occur in future runs; that better control of consistency can be expected to reduce the variability in production of basis weight results? We would be almost certain if the results in each column varied no more than, say, 0.05 from the average of that column; and we would be equally sure the effect was not real if the individual results varied as much as 1.0 lb from their column average. So somewhere between these extremes must reside a lack of within-column repeatability which, compared to the difference among column averages, represents a 50% chance the effect is real. Statistical tables give values of a ratio of that type which can occur, say, 5% of the time by chance; and another ratio which can occur 1% of the time. If the experimental ratio falls between this pair of tabulated ratios, we might conclude that the probability the effect is real is better than 95, but not as good as 99%.

But we still cannot conclude validly that we thus have a result statistically significant at the 0.05 level. We must have taken experimental procedure precautions to prevent variables other than headbox consistency (even unsuspected ones) having a bias, or sufficiently serious unequal effect, upon the column averages. These variables are possibly in the material being tested (the furnish) or factors that change with time during the experiment (in the paper machine, mill environments, machine tenders' actions, testing equipment, or laboratory environments).

The precautions simply force all other factors, which vary with test material and with time, to show their total influence on the effect (by increasing the within-column variability); and virtually to balance out throughout all the data so that they have substantially the same unbalanced effect within columns as between columns. The extent to which the unbalanced effect can falsely indicate a column-to-column difference is allowed for in the previously mentioned table of ratios at given probability levels. All other factors will act this way when the assignment of test material and when the time sequence of testing are separately randomized.

A second factor can be "crossed" with headbox consistency, for example, wire speed in this experimental design—and, say, these were the basis-weight results:

	C_L	C_M	C_H	Av.
WS_L	32.4	32.9	33.4	32.8
	32.6	32.6	33.1	
WS_H	30.7	31.8	32.4	31.7
	30.8	32.1	32.4	
Av.	31.6	32.4	32.8	

The main effects of consistency can now be seen separately from the main effects of wire speed; the differences among vertical averages show the former, and that between the pair of horizontal averages shows the latter. Such ability to separate main effects comes from the exact balance property of this design: at each level of consistency the contribution of the two wire-speed influences is alike, and at each level of wire speed, the contribution of the three consistency influences is alike.

To see another property of this design (called a full-factorial), which has every combination of consistency and wire speed of the experiment represented in the six cells, visualize a graph of two curves from these results. The plot would be of basis weight on the vertical scale against the three levels of consistency on the horizontal scale. The top curve would be labeled "low wire speed"; the bottom, "high wire speed."

These cell average results show the points through which the two curves would pass:

	C_L	C_M	C_H
WS_L	32.5	32.8	33.3
WS_H	30.8	32.0	32.4

Note that while the difference between the two curves at C_M and at C_H are about the same, 0.8 and 0.9, at C_L the difference is 1.7. The two curves are not parallel. Again a ratio is formed from these results to compare against tabulated ones to see if this nonparallelism (called an interaction between consistency and wire speed) is statistically significant. The numerator is a measure of the cell-to-cell variability (corrected for the column-to-column and for the row-to-row variability), while the denominator is a measure of the individual reading-to-reading variability within cells. This latter term properly represents a measure of the total effect of all other variables present, although unidentified, which were operating during the conduct of the experiments; because material and time sequence were both randomized.

PRACTICAL PROBLEMS GROW AS THE NUMBER OF VARIABLES INCREASES

While it is a simple matter to include still other factors in a full-factorial experimental design, the number of combinations to be run grows. It is the multiplication product of the number of levels for each factor. Fractional factorials have been developed to cut down the number of combinations tested, but still maintain an exact balance so that desired effects will be statistically separable; the contributions of other such effects upon the one being analyzed being alike, thus being neutralized or balanced out. They may call for $^1/_2$, $^1/_8$, or up to, say $^1/_{256}$ of the number of cells of the corresponding full-factorial design. Unfortunately, the price paid for such a reduction of tests is the complete confounding (cannot be separated statistically) of main effects and interactions with others; the greater the fractional reduction (used with the larger numbers of possible combinations) the greater is the loss of possible information. Proponents of the use of fractional factorials say, "in many experiments interactions involving three or more factors can be considered negligible."

The goal of this paper is to show how to *discover* the factors to be tested, and so lead to substantial—rather than minor—improvements in processes and products. Advance knowledge is needed for fractional factorial planning: certain effects are not expected to be important. This very knowledge is absent when one embarks upon an objective search for the unknown.

To show the necessary modification in thinking in applying statistical experimental design principles, a case study should aid in serving as a vehicle for developing what might be termed a heuristic philosophy—one serving to discover or to stimulate investigation. The case study is a synthetic one, made

up of a combination of certain experiences from out consulting work. The names are changed, but the statistical situations are individually real. Our hope is to present the analytical sequence clearly enough to suggest an approach to get you started on a practical program to bring about economically significant improvements.

CASE STUDY

The Real Strong Paper Co. was the recipient of complaints from two important customers; for their applications the specified nominal strength was adequate, but too many failures were being encountered at inappropriate times. The paper just did not have sufficiently uniform bursting strength.

The philosophy of the approach for finding unknown causes follows this logic:

1. Relatively few, not many, causes are responsible for the quality difficulty.

2. These causes, having a controlling influence, are not constant in their action; thereby causing the quality characteristic to vary.

3. The vital few causes can be identified by a rapidly converging, process-of-elimination procedure (like the parlor game of 20 questions); but using the *relative* amounts of variation among "components of variability" as clues as to where to go next.

No time was spent on thinking what might cause a lack of uniformity in strength, since the list could be virtually endless, ranging from mill variables which could cause a variation in formation and in fiber length to nature's variables which change the environments associated with wood growth. Instead, step one of the variation research method (so named by one of our clients) is to allow the variation in quality to show itself in such a way that it can be separated quantitatively into components of variation—in the same manner that the 20 question strategy determines whether the unknown object is animal, mineral, or vegetable. The largest component of variation must have associated with it a cause or causes of such variation. If the difficulty now, with this clue, can be turned on and off like water with a faucet, a controlling cause certainly has been isolated. If it cannot, the procedure asks a second objective question, thus homing in closer on the elusive cause.

Bursting strength test results were compared (calculating a *difference* each time) to get these components of variation: (1) within reel, cross direction; (2) within reel, machine direction; (3) reel to reel; (4) batch to batch of furnish; (5) shift to shift, and (6) day to day.

The bursting strengths from the first two of these components in a single reel were averaged. That average was compared with a similar number from each of successively produced reels to get component no. 3. These averages from several reels made from one batch of furnish were averaged to get a single number for that batch. It, compared with similar numbers for other batches, showed the size of component no. 4. Several batch averages gave the shift average; and the average of three shifts gave the day average.

Revelation no. 1: the batch-to-batch differences were by far the largest! So now thoughts were concentrated on mill variables; what happens in the mill that could make one batch so different from another? Variables were mentioned which were associated with the headbox, beater, clear back to loading the digester. Still too many to think about testing a few to see if they are important. Question no. 2 could well be designed to isolate the operation which first introduces the batch-to-batch difference.

The complications of routing pulp back and forth between a pair of parallel series of processing steps for this elimination procedure were discussed. A full-factorial design could be expected to show that one of a pair of similar steps introduced more batch-to-batch difference than the other; or that an interacting combination of operations was responsible.

Instead of measuring bursting strength directly, it was suggested that the transfer of pulp between lines could be avoided by measuring pulp freeness at each operation, it being a property that would be expected to correlate with several properties of finished paper. First it had to be confirmed that batch-to-batch freeness in the headbox varied more than within-batch, shift-to-shift, and day-to-day freeness. It did.

Working back, step by step, this large batch-to-batch difference in freeness was found to persist clear back to the blow pit. Revelation no. 2: something in the digester made the freeness vary. Now the number of associated factors was becoming smaller. A statistically designed experiment could be expected to show the effect on paper bursting strength of cooking temperature and time, steam pressure, proportion of different wood species and of cooking liquor to wood chips, digester to "similar" digester differences, or whether the randomized contribution of some other factors (called residual variation) was still the largest. Changes of level had to be made in each factor to see which one or which combination controlled paper strength. The plans were to test these indicated numbers of levels within a range representative of current mill operating practice so as to produce saleable paper throughout the "experiment":

Factor	*Levels*
Cooking temperature	Several (it would be recorded for each test batch)
Cooking	3
Steam pressure	2
Proportion of wood species	6
Proportion of cooking liquor to wood weight	3
Among digesters	4

Without cooking temperature, the product of these levels indicates 432 possible combinations, or cells of a full-factorial design. Beyond being an impractical number of combinations to test, the cooking temperature was difficult to control independently of the other factors. So it was planned to run a random sample of 30 of the 432 combinations to be statistically representative of the entire population. The resulting burst strength for the 30 batches would then be plotted separately against the cooking temperature recorded for each, against the three cooking times used, the two steam pressures, etc.

Clear differences in strength were associated with the two kinds of proportions used as factors, with cooking temperature and with time. Steam pressure and unknown digester differences had almost no effect. The residual variation was small. The close limits to which either proportion or the cooking temperature would have to be controlled clearly would be impractical without major improvements in and additions to digester equipment. But a plot of blow pit freeness against cooking time suggested a practical approach.

A tap was added to the side of the digester so that a beaker of pulp could be drawn before the end of the cooking cycle. Its freeness was found to be related to the blown pulp freeness. So the above plot could be used to indicate, for each digester load how much longer it should be cooked to result in pulp having closer to the desired blown freeness. Thus an important controlling factor was isolated by the process of elimination and a final paper quality variation was substantially reduced.

Lest there be any misunderstanding, the message we are trying to convey by this synthetic story is *not* that this particular "solution" is the way to control bursting strength. We are trying to illustrate, without revealing confidences, that a process-of-elimination method, aided by appropriate statistically designed experiments can be expected to improve your processes and product substantially.

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A Study of the Variables Affecting the Viscosity and Adhesive Strength of Pigmented Paper Coatings

L. E. GEORGEVITS and W. L. MARINO

Subcommittee IV, Section A of ASTM Committee D-25, has been studying test methods for casein and isolated soy protein as related to their use in paper coating. Two of the most important properties under study are viscosity and adhesive strength. This study reviews the effect of temperature, per cent solids, adhesive ratio, pH, alkali, coat weight, paper, drying conditions, and mixing conditions on viscosity and adhesive strength. Cooperative tests were made with 12 laboratories studying proposed methods for determining the viscosity of a 15% casein solution and the viscosity of a clay coating at 45% solids with a 15% adhesive ratio. Drawdowns of the pigmented coating were made and each participating laboratory determined the adhesive strength on the drawdowns made by all the laboratories, using the Dennison wax pick test and in some cases the I.G.T. printability tester. Poor correlation was obtained between laboratories regarding the viscosity of the casein solution. The average viscosity was 1401 cp. with a standard deviation of 539 cp. Better results were obtained on the pigmented coating with a viscosity at 100 r.p.m. of 700 cp. and a standard deviation of 100 cp. The average wax pick value was 9.4 with a standard deviation of only 0.23. The I.G.T. results were very scattered.

THE work reported in this paper was done to study the possible sources of error in proposed test methods prepared by ASTM Committee D-25 Subcommittee IV A for determining the viscosity and adhesive strength of casein and isolated soy protein in a pigmented paper coating. The work done by Committee D-25 was on both casein and isolated soy protein but in this paper we will limit our discussion to only casein. The data on both the casein and isolated soy protein were sent out as a special report to all members of the subcommittee in February, 1961.

Committee D-25 was organized by ASTM in June, 1956, its scope was the "Definitions of terms and the study and formulation of test methods and specifications covering industrial casein, isolated soy protein, and similar proteinaceous materials." As a first step, before other tests could be developed, a sampling procedure was required. This procedure has been adopted as Tentative Method D 1697–59 T. The work of the committee now centered about the preparation of standard chemical and physical tests.

CHEMICAL TESTS

The chemical tests were prepared with little difficulty using accepted analytical procedures in most cases. In June 1960 Subcommittee III on Chemical Tests issued the following tentative test methods.

D 1797	Ash in Casein and Isolated Soy Protein
D 1798	Fixed Ash in Casein and Isolated Soy Protein
D 1800	Alkali Requirement of Casein and Isolated Soy Protein
D 1801	Free Acidity in Casein and Isolated Soy Protein
D 1802	Moisture in Casein and Isolated Soy Protein
D 1803	Nitrogen in Casein and Isolated Soy Protein
D 1804	Oil in Casein and Isolated Soy Protein

Subcommittee III is now studying test methods for fat content and bacteria count.

PHYSICAL TESTS

While things were going very well for Subcommittee III, Subcommittee IV on Physical Tests was having considerable difficulty preparing test methods that were reproducible. The first two physical tests to be studied were those for dry particle size and viscosity. Proposed test methods were prepared and samples of caesin and isolated soy protein were sent to 11 laboratories. In two series of tests it has been difficult to obtain good agreement between laboratories regarding particle size analysis, particularly when fines were present. A special subcommittee is now studying a revised particle size test using brushing in combination with a shaking method. Other physical tests under study are those for cleanness, odor, insolubles, and foaming tendency.

Viscosity Test

The problems in setting up a viscosity test suitable to all concerned were quite difficult because of the wide range of end uses for casein. The total usage of casein for industrial purposes in 1960 was approximately 70 million lb. and for isolated soy protein, approximately 45 million lb. The major applications in order of their importance are in paper coatings, powdered paints and joint cements, adhesives, and as a thickener or stabilizer in latex paints, leather finishes, and shoe dressings.

Since paper coating was the major application it was decided to set up the initial test around a typical paper coating formula. This in itself caused considerable trouble because pigmented paper coatings vary from 35 to 60% solids, and are prepared under a wide variety of conditions.

In setting up a viscosity test on the adhesive there were five important variables. (A) type equipment, (B) solids content, (C) type and amount of alkali, (D) cooking temperature, and (E) type viscometer.

A. The committee readily agreed that a variable speed, heavy duty, propeller mixer was satisfactory. However, it has been very difficult to define such a mixer closely. Surprisingly enough, few ASTM or TAPPI committees require the use of a mixer and no specification has been set up. Such a specification is now under consideration by ASTM.

B. Solutions of casein or isolated soy protein differ widely in viscosity, therefore there was considerable discussion as to what solids content should be used in preparing the adhesive solution. Solutions of both are usually prepared at 10 to 30% solids. Generally speaking, the lower the viscosity the easier it is to obtain reproducible results. It was therefore decided to work at 15% solids for both the casein and isolated soy protein.

C. There is a wide range as to the type and amount of alkali used to prepare solutions of either casein or isolated soy protein. Combinations of two alkalies are quite common. The alkalies most widely used are, in order of importance, ammonium hydroxide, borax, sodium hydroxide, soda ash, and lime. Of these ammonium hydroxide was chosen because slight variations in the amount of ammonium hydroxide used have the least effect on viscosity. By using an excess amount of ammonium hydroxide, variations in the alkali requirement of the casein could be eliminated.

D. In preparing solutions of casein the cooking temperature varies widely. In joint cements and caesin paints the casein is cut at room temperature averaging 60 to 80°F., but at times 40°F. The latter condition is a special case we plan to study in the future. In some cases casein solutions for paper coating are prepared at room temperature but in most cases solutions are cooked at 120 to 140°F. For paints and adhesive work where long term stability is required it is very important to cook at 170 to 180°F. to destroy any naturally occurring enzymes. The enzymes present cannot be destroyed chemically and if the solution is not cooked at a temperature above 170°F. it will thin out rapidly on standing 1 month even if sufficient preservative is present to prevent spoilage. Since we decided to develop the initial tests for the use of casein in paper coating a cooking temperature of 130°F. for 30 min. was decided upon.

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E. Regarding viscometers, there were four types commonly used by the committee members, the Brookfield, Stormer, Ford cup, and Bloom pipet. All subcommittee members had the Brookfield, therefore, it was adopted. However, the Brookfield is made in four widely used models having different speeds of rotation. Since this was only preliminary work each laboratory was requested to use the model on hand; but the RVF model was preferred.

Table I. Results of Cooperative Tests on Determining the Brookfield Viscosity in Apparent Centipoise on a 15% Solid Casein Solution at 25°C. (77°F.)

(Tests made June, 1957)

	Speed, r.p.m.			
	6	12	30	60
Lab. No.	Model LVF			
A	1300	1220	1188	1172
A	1320	1330	1268	1240
B	1100	1100	1120	1112
B	1000	1050	1080	1072
C	1600	1500	1420	1400
C	1500	1400	1300	1260
D	1700	1550	1500	1450
D	1600	1550	1480	1440
E	1090	1140	1144	1146
E	1120	1120	1142	1112
F	1680	1670	1634	1590
F	1600	1600	1584	1560
	10	20	50	100
Lab. No.	Model RVF			
G	940	950	964	981
G	970	1000	996	1020
H	1300	1300	1370	1350
H	1200	1200	1220	1210
I	1400	1400	1420	1400
I	1300	1300	1300	1320
J	1200	1200	1240	1220
K	1000	1000	1028	1030
K	1100	1150	1188	1164
	2	4	10	20
Lab. No.	Model RVF			
L	1120	1120	1112	1104
L	1240	1220	1232	1228
M	1480	1400	1376	1310
N	800	900	1040	1070
N	900	970	1170	1170
	3	6	12	30
Lab. No.	Model LVF			
O	1000	1000	987	975
P	910	875	862	845
Average viscosity			1223 cp.	
Standard deviation			211 cp.	

The results of these first cooperative tests on casein made in 1957 are shown in Table I.

In studying these results it was found that the solutions were almost Newtonian in nature and therefore the average of all readings on all viscometers were taken. The casein solution had a viscosity of 1223 cp. with a standard deviation of 211 cp.

These results were not satisfactory, therefore, a second series of tests was made. It was thought that part of the variation might be due to the viscometers themselves, therefore, samples of oil with a calibrated viscosity of 109 and 1199 cp. were sent out to all subcommittee members. A close correlation was obtained on these, indicating the viscometers were accurate enough but the problem was in preparing the adhesive solution. In the second series of cooperative tests, a few minor changes in procedure were made and viscosity readings were taken at 130°F. immediately after cooking, then the solutions were cooled and viscosities taken at 77°F. The results at 77°F. are shown in Table II. The average viscosity was 1118 cp. with a standard deviation of 388 cp. There are again wide variations in results. Some of the reasons for this variation in results will be discussed later.

PIGMENTED COATINGS

Early in our committee work a list of important physical properties was prepared. Heading this list was (1) viscosity in a pigmented coating and (2) adhesive strength or pigment binding ability. When the second series of viscosity tests was made, a small task force with four members made a few preliminary tests regarding the preparation of a pigmented paper coating.

Coatings of casein or isolated soy protein with no latex present are usually prepared at 35 to 55% solids using 10 to 20% adhesive. It was decided to work at 45% solids using a 15% adhesive ratio. The same casein solution used for determining viscosity was used in preparing the coating. In the initial tests samples of casein and isolated soy protein were sent out together with the raw stock to be used. There was considerable controversy in deciding how to select a standard raw stock. All coating people agree that the raw stock is very important in influencing the properties of the coating. Since a large portion of the casein and isolated soy protein is used for off-set and label paper it has been decided to use a label grade groundwood-free coating raw stock prepared by the Newton Falls Paper Co. This sheet has very high internal strength and is not two sided. In this early work the type of clay to be used was not specified and was merely defined as a coating grade clay. The four people on the task group chose three different clays. When the same clay was used a fairly close agreement was obtained regarding viscosity and adhesive strength, but when different clays were used there was a wide range of results. Drawdowns were made with these coatings using an 0.001 in. Bird film applicator and drying for 60 sec. at 200°F. The results are shown in Table III.

Table II. Results of Cooperative Tests on Determining the Brookfield Viscosity in Apparent Centipoise on a 15% Solid Casein Solution at 25°C. (77°F.)

(Tests made June, 1959)

Co.	Spindle no.	Speed, r.p.m. 10	20	50	100
		Model RVF			
B	4	1880	1880	1880	1886
E	4	1300	1300	1230	1200
H	3	850	850	854	875
I	5	822	508	485	453
		6	12	30	60
		Model LVF			
A	3	1050	1050	1060	1060
D	2	1120	1020	1260	1220
G	3	1395	1328	1288	1303
G	3	1122	1108	1109	1171
		2	4	10	20
		Model RVF			
J	1	810	806	808	810
F	2	600	600	588	594
Average viscosity				1118 cp.	
Standard deviation				388 cp.	

A new series of cooperative tests was set up and samples of a predispersed KCS clay slip (Georgia Kaolin Co.) at 70% solids were sent out together with samples of casein, isolated soy protein, and coating raw stock.

Adhesive Solution

The adhesive solution was again prepared at 15% solids

with 12% NH_4OH heating at 130°F. as before. Everyone was cautioned to be as careful as possible in all weighings and temperature readings to eliminate these variables. In this series of tests 12 laboratories participated. The results of these tests are shown in Table IV. In this case the average

Table III. Comparison of pH, Viscosity, and Adhesive Strength of Paper Coatings with Casein at 45% Solids and 15% Adhesive Ratio

	Company			
	A	*B*	*C*	*D*
Type clay	1	1	2	3
Coating color				
pH	9.3	9.3	9.4	9.5
Viscosity, cp., 25°C.				
10 r.p.m.	985	1500	2180	860
20 r.p.m.	835	1225	1580	740
50 r.p.m.	588	830	1036	520
100 r.p.m.	488	635	706	430
Wax pick	10	10	11.2	8.7

Company	*Type mixer used*
A	Heavy duty stirrer
B	Waring Blendor
C	Hamilton Beach
D	Heavy duty stirrer

viscosity was 1401 cp. and the standard deviation was 539 cp. This large standard deviation was obtained because the viscosity on the solutions varied from 855 to 2800 cp. and none of the values were far enough out of line to discard.

Viscosity of Pigmented Coatings

The coatings were prepared by first adding 41 parts of water and 8 parts of a 10% NaOH solution to 572 parts of the 70% solids clay slip. To the pigment slip was then added, 400 parts of the 15% solids adhesive solution. High speed agitation was used when first adding the casein solution, but

Table IV. Results of Cooperative Tests on Determining the Brookfield Viscosity in Apparent Centipoise on a 15% Solid Casein Solution at 25°C.

(Tests made August, 1960)

Laboratory no.	*Speed, r.p.m.*			
	10	*20*	*50*	*100*
	Model RVF			
1	865	862	868	855
2	1410	1405	1400	1356
4	2800	2700	2650	2520
6	1100	1050	1040	1040
7	1190	1195	1206	1260
8	1480	1450	1446	1468
9	1180	1230	1260	1266
12	1360	1253	1185	1233
	6	*12*	*30*	*60*
	Model LVF			
5	1275	1250	1187	1175
11	1120	1070	1048	1030
	2	*4*	*10*	*20*
	Model RVF			
3	2400	2400	2440	2420
10	1000	950	960	950
Average viscosity	1401 cp.			
Standard deviation	539 cp.			

then the speed was reduced to prevent excess foaming. When the casein solution is first added to the pigment slurry some initial thickening may occur and if the stirrer is not of the heavy duty type some flocculation may occur. Good results have been obtained using a heavy duty air stirrer in place of an electric mixer.

The viscosity data on the pigmented coatings prepared by the 12 laboratories are shown in Table V.

In tabulating these data we calculated the standard deviation on only the results using the RVF Model Brookfield which eight laboratories used. We obtained at 50 r.p.m. an average viscosity of 950 cp. with a standard deviation of only 132 cp. This was quite surprising in view of the large standard deviation on the casein solution. It shows that the method as now written is acceptable as a standard procedure for preparing a pigmented casein coating.

Adhesive Strength

The most widely used method for determining adhesive strength is the Dennison wax pick test. This method is described in TAPPI Standard T 459 m-48. Modifications of this test are described in TAPPI Routine Control Methods RC-6 and RC-61. It was decided to use procedure T 459 m-48 for determining the comparative adhesive strength of the above coatings. In the past few years there has also been considerable interest in the use of the IGT pick tester for determining adhesive strength. Lehigh University has done considerable work on surface strength evaluation under

Table V. Results of Cooperative Tests on Determining the Brookfield Viscosity in Apparent Centipoise of a 45% Solid Casein Coating with 15% Adhesive Ratio at 25°C.

(Tests made August, 1960)

Laboratory no.	*Speed. r.p.m.*			
	10	*20*	*50*	*100*
	Model RVF			
1	1735	1255	801	603
2	1820	1540	1072	732
4	2050	1450	1215	927
6	1650	1350	900	650
7	1680	1425	1000	746
8	1360	1205	848	650
9	1430	1245	876	649
12	1480	1250	892	638
Average viscosity	1650	1340	950	700
Standard deviation	216	113	132	100
	6	*12*	*30*	*60*
	Model LVF			
5	5050	3450	1890	1225
11	2220	1820	1256	910
Average viscosity	3635	2635	1573	1068
	2	*4*	*10*	*20*
	Model RVF			
3	2600	2500	2140	1670
10	2550	2350	1780	1300
Average viscosity	2675	2425	1960	1485

TAPPI Research Projects 130 and 166. They have prepared "Routine Control Instructions for Pick Measurements on the I.G.T. Printability Tester." An advance copy of this procedure was sent to the members of the subcommittee and those having an IGT pick tester were asked to make tests on this instrument as well as wax pick tests. In preparing their procedure Lehigh emphasized that this method does not represent the ultimate "standard method" but is written as an aid to those already using the IGT tester in quality control programs. There are still a number of variables in the use of the IGT pick tester that are under study.

Results of Cooperative Tests

After the 12 cooperating laboratories made the coatings described above they each made 12 drawdowns on the standard raw stock. They each sent us the complete set and we sent out a set of 12 drawdowns, one made by each laboratory, to the same cooperating laboratories. In addition some

commercially coated paper (all casein) obtained from the Newton Falls Paper Co. was sent out and coded as sample 13.

All of the laboratories did not complete this portion of the tests, but the results of the wax pick test from 11 laboratories are shown in Table VI.

We first determined the coat weight applied by the 12 labs. The raw stock had a basis weight of 46 lb. (25 × 38—500), and the average coating applied was 16.7 lb. This was a little high, but if a drawdown blade of less than 0.001 in. in thickness is used it is difficult to apply a uniform thickness.

adhesive ratio, pick will generally be obtained with a no. 4 ink.

ADHESIVE SOLUTION VARIABLES

After reviewing the above data we decided to study the possible causes of error that could give us such erratic results.

To start with we studied the adhesive solution itself. In all of the following work the same blended sample of 30-mesh New Zealand lactic casein was used as the adhesive unless shown otherwise.

Table VI. Wax Pick and IGT Pick Strength Determinations; on Samples of Casein Coated Paper Prepared by 12 Laboratories

	Coating													
	1	*2*	*3*	*4*	*5*	*6*	*7*	*8*	*9*	*10*	*11*	*12*	*13*	*Av.*
Coat wt. per ream, lb.	4.3	19.6	16.4	20.6	13.8	17.7	19.6	16.1	15.2	15.2	10.7	15.0	—	16.8
Critical Wax Pick Values														
Lab. no. 3	9	10	10	8	9	9	9	9	9	9	8	10	9	9.0
4	10	10	10	10	10	10	10	10	9	8	9	9	10	9.6
5	10	10	10	9	9	10	10	10	9	9	9	10	9	9.5
6	9	9	9	9	9	9	8	9	9	9	8	8	10	8.8
7	9	9	9.3	8.7	8	9	9	9.3	8	7.7	8.3	8.3	8.7	8.6
8	11	..	11	11	..	11	11	11	12	..	12	..	10	11.1
9	10	9.3	10	8.7	8.7	9	9.3	9.3	9.7	9.3	9	9.3	9	9.2
10	10	10	10	10	10	10	10	9	10	10	10	9	8	9.7
11	10	10	10	10	9	10	10	10	9	10	10	10	10	9.8
12	9	9	10	9	9	10	10	10	10	9	9	10	10	9.5
14	9	9	10	10	9	9	9	9	9	9	9	9	9	9.1
Average	9.6	9.5	9.9	9.4	9.1	9.6	9.5	9.6	9.4	9.0	9.9	9.2	9.3	9.4
	Standard deviation 0.23													
IGT Values in cm./sec. Using No. 4 Ink, Spring Drive, Speed B, 50 Kg. Pressure														
Lab. no. 4	206	114	114	108	110	100	108	140	132	158	162	136	227	139
5	269	180	137	141	150	105	140	145	180	147	145	148	212	161
8	..	184	..	..	149	..	..	..	..	162	..	..	155	162
11	202	127	153	142	127	122	163	150	193	147	184	153	168	156
14	230	174	214	178	183	170	206	236	224	162	240	188	186	199
Average	226	156	154	142	144	124	154	167	182	155	182	156	189	163
IGT Values in cm./sec. Using No. 5 Ink, Spring Drive, Speed B, 50 Kg. Pressure														
Lab. no. 2	251	162	133	118	138	..	168	..	193	158	168	..	..	165
IGT Values in cm./sec. Using No. 4 Ink, Spring Drive, Speed A, 50 Kg. Pressure														
Lab. no. 6	183	73	90	106	90	73	73	106	90	90	133	52	106	97
IGT Values in cm./sec. Using No. 6 Ink, Pendulum Drive, 50 Kg. Pressure														
Lab. no. 1	90	47	52	56	63	61	80	76	73	60	69	84	74	68

The use of an 0.00075 in. blade should be considered in the future.

The wax pick values came out surprisingly good. The coatings had an average wax pick of 9.4 with a standard deviation of 0.23 and the range on the drawdowns prepared by the 12 laboratories was only 9.0 to 9.9. This indicates the coating has a rating of 9 or 10. In this work we asked to report the highest wax pick number at which no pick of any type was obtained. In our own work we found a variation of from 9 to 10 could readily be obtained due to a variation in wax procedure. It is noted that in running the pick tests lab no. 8 consistently reported a high value while lab no. 7 consistently gave a low value. Part of our problem may be in judging the end point, and we are going to have all the laboratories return their tested samples to us for evaluation. It has also been suggested that in future work a study be made using the amount of binder required to obtain a desired wax pick value, as the end point.

It is much more difficult to draw any conclusions on the IGT tests because the same conditions were not used in all cases. However, the results seem more erratic than with the wax pick. We will await further work from Lehigh before adopting this procedure.

In our own work studying the variables in the IGT test using control sheet 13, we found that pick seldom occurred with a no. 3 IPI ink, and the paper always showed very severe picking with the no. 5 ink. Therefore, the test does indicate that with this particular casein at 45% solids and a 15%

Spindle Size and Temperature

The effect of spindle size on viscosity was first investigated. The results are shown in Table VII. The results clearly show that spindles 1, 2, and 3 were too large. Spindle 6 too small but either spindle 4 or 5 were satisfactory, with 4 preferred. The results using different spindles checked out quite well except at 10 r.p.m. Our findings have been that the readings at 10 r.p.m. can be quite misleading in their significance. This is particularly shown in Table V where the greatest standard deviation occurred at 10 r.p.m.

The next variable studied was the effect of spindle temperature on viscosity. Using the 4 spindle it was placed in water of various temperatures and adjusted to a temperature of 60, 80, and 100°F. In all cases the solution was adjusted to 77°F. The effect of spindle temperature on viscosity is shown below.

Spindle temperature, °F.	*Viscosity, cp.—50 r.p.m.*
60	1540
80	1440
100	1320

These figures show that it is quite important to bring the spindle to the solution temperature before taking a reading.

Effect of Temperature

In Fig. 1 is shown the effect of temperature on the vis-

cosity of a 15% casein solution and 45% solids coating. It can be seen that a slight change in temperature can cause a great change in viscosity. This is probably one of the greatest sources of error in the test. It may be necessary to keep the solution in a constant temperature bath while the viscosity reading is being made. Another major source of error is in the thermometer. While a thermometer meeting

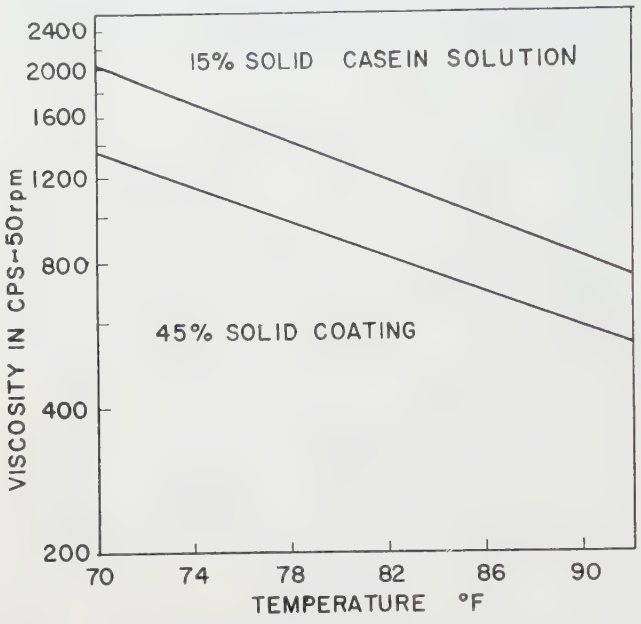

Fig. 1. Effect of temperature on the viscosity of a 15% solid casein solution and 45% solid coating

ASTM Specification E-1 is called for, our tests indicated that some old thermometers in our laboratory were off as much as ±2°F. In running any future viscosity tests all laboratories should check the accuracy of their thermometers.

Effect of Time

A second source of error is the time factor, that is the elapsed time between cooling the solution and taking the viscosity reading after adjusting to the proper temperature. The test method calls for taking the viscosity 1 hour after bringing the solution to 77°F. The effect of time on viscosity is

Table VII. Effect of Spindle Size on Viscosity of 15% Solid Casein Solution at 25°C.

(—Brookfield Model RVF Speed, r.p.m.)

Spindle		*Speed, r.p.m.* 10	20	50	100
1	Scale reading	Off	Off	Off	Off
	App. Viscosity, cp.	..	..	..	..
2	Scale Reading	32.5	65	Off	Off
	App. Viscosity, cp.	1300	1300	..	..
3	Scale Reading	14	27	68	Off
	App. Viscosity, cp.	1400	1350	1360	..
4	Scale Reading	7.5	14.5	35	68.5
	App. Viscosity, cp.	1500	1450	1400	1370
5	Scale Reading	4	7.5	18	34.5
	App. Viscosity, cp.	1600	1500	1440	1380
6	Scale Reading	2	3	7.5	14.5
	App. Viscosity, cp.	2000	1500	1500	1450

shown in Table VIII. This appears to be a minor source of error.

Solids Content

A third source of error is the concentration of the solution. The test method calls for adjusting the solution back to tare weight to make up for any water lost during cooking. The effect of solids on viscosity is shown in Fig. 2. Since there is

Table VIII. Effect of Holding Time After Cooling to 25°C. on the Viscosity of a 15% Solid Casein Solution

Time, hr.	0.1	0.5	1.0	2.0	4.0	24.0
Viscosity, cp. 50 r.p.m.—25°C.	1260	1460	1580	1400	1320	1420

the possibility of a slight error in weighing out the chemicals or water this may easily result in a considerable error in viscosity. A possible source of error here may be due to a change in the moisture content of the casein. A change of 3% in the moisture content of the casein will change the concentration of a 15% solid solution by 0.5%. Solutions of the same casein were prepared on two different days, one day with high humidity and one with low humidity. The casein was first spread out and allowed to reach equilibrium, and the moisture content determined. The results are shown below:

Moisture on casein, %.....	7.8	10.4
Viscosity, cp. 50 r.p.m. 77°F.....	1620	1140

To compensate for this possible error in our proposed method the moisture content of the casein is taken and the amount of casein used is adjusted so that it is equivalent to a 9% moisture content casein, or 91 g. of moisture-free casein.

Effect of Viscometer Type

In our laboratory we have four different Brookfields available. The same casein solution was tested on the four instru-

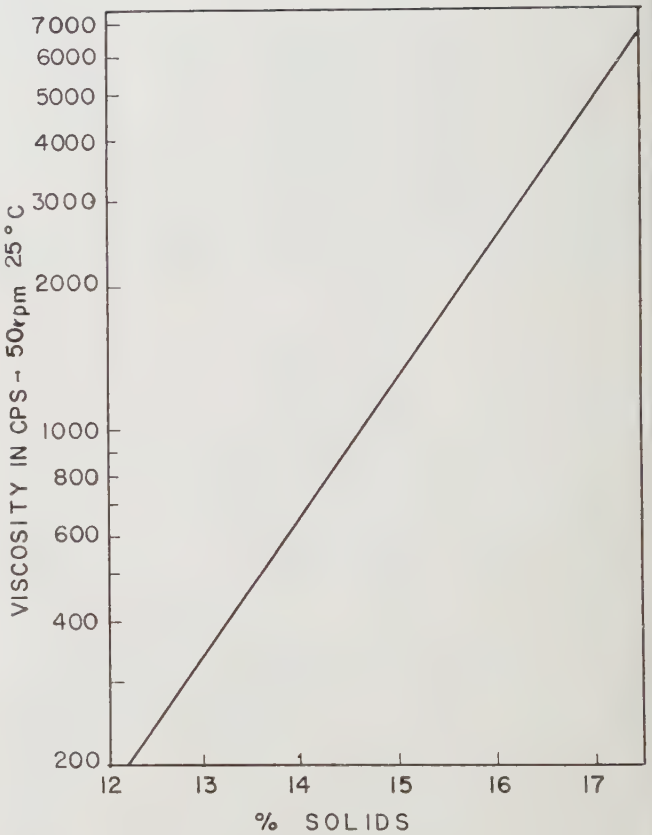

Fig. 2. Effect of solids content on viscosity of a casein solution

ments. The results are shown in Table IX. The results indicate that the large variation in viscosity obtained between laboratories is not due to an error in the Brookfield viscometers.

In the course of our work, solutions of the same casein were prepared a number of times by two different technicians using

Table IX. Comparison of the Viscosity of the Same 15% Solid Casein Solution Using Six Different Brookfield Viscometers

Instrument	*Spindle*	*Speed, r.p.m.* 2	4	10	20
		Model RVF			
A	2	1500	1450	1460	1450
B	2	1600	1600	1600	1600
C	2	..	..	..	1500
D	2	1550	1550	1540	1530
		6	*12*	*30*	*60*
		Model LVF			
E	3	1500	1450	1440	1440
		10	*20*	*50*	*100*
		Model RVF			
F	4	1400	1400	1400	1410

the same basic equipment. The viscosities of these solutions are shown in Table X. This variance may be due to any of the variables described above.

Factors Affecting Coating Viscosity

A study was next made of the variables effecting the viscosity of the coating. In this work we prepared our own 70% solid slurry of KCS clay mixing the clay with 0.3% Calgon for 15 min. at high speed on a Cowles Dissolver. The coatings in all this work were prepared using a heavy duty variable speed air stirrer with a circular propeller having a diameter of $1^1/_2$ in. Unless otherwise specified the coatings are all at 45% solids with a 15% adhesive ratio.

Effect of Temperature

In Figure 1 the effect of temperature on the viscosity of the coating is shown. Drawdowns of this coating were made with

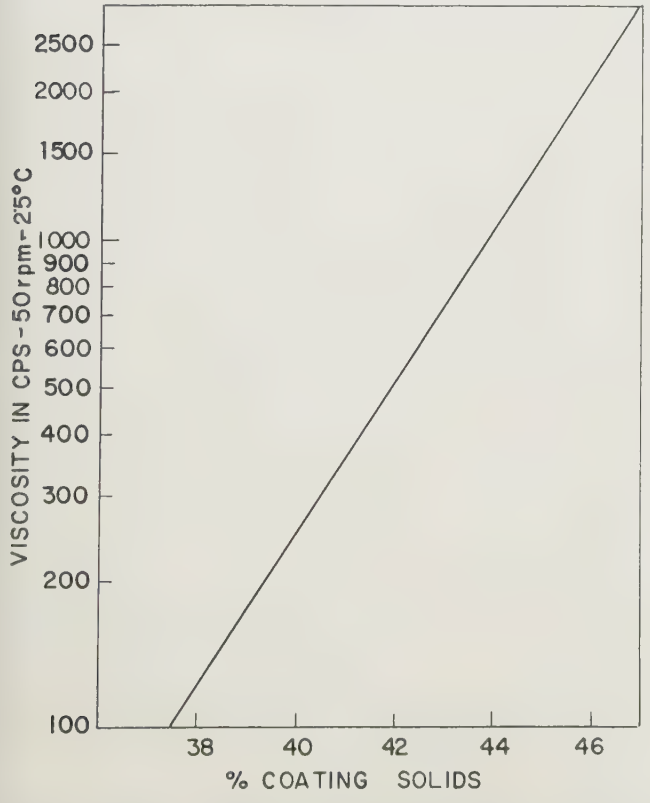

Fig. 3. Effect of solids content on viscosity of casein coating with 15% adhesive ratio

Table X. Comparison of the Viscosity of a 15% Solid Casein Solution Made at Seven Different Times in the Same Laboratory with the Same Casein

Speed, r.p.m.	*Brookfield viscosity, cp.* A	B	C	D	E	F	G
10	1500	1300	1400	1400	1200	1700	1800
20	1450	1250	1350	1400	1200	1650	1750
50	1440	1240	1300	1400	1200	1640	1720
100	1440	1220	1270	1380	1230	1610	1680
pH	9.4	9.6	9.5	9.3	9.3	9.4	9.4

the coating at 70, 80, 90, and 100°F. No significant difference in adhesive strength was found as the temperature varied.

Effect of Solids

In Table XI is shown the effect of coating solids content on viscosity, wax pick, and I.G.T. pick strength. The viscosity is a direct log function of solids content as shown in Fig. 3. The wax pick and I.G.T. strength did not vary significantly as solids changed.

Effect of pH and Cooking Temperature

The effect on pH, viscosity, and adhesive strength when using various amounts of ammonium hydroxide to cut the casein is shown in Table XII. It is seen that a change in ammonium hydroxide content from 8 to 14% increases the pH from 8.9 to only 9.4, and changes the coating pH from only 9.1 to 9.7 with only a slight effect on viscosity. Changes in the amount of ammonium hydroxide from 4 to 14% have very little effect on adhesive strength because the casein is dissolved in all cases. There have been statements made that casein has the maximum adhesive strength at pH 9.0. The reason for this statement is that poor grades of casein are difficult to dissolve at pH 7.0 or lower and in the past mills found it best to work at pH 9.0. If the casein is completely dissolved it should have equal adhesive strength at pH 6.0, 9.0, or 11.0. The objection to working at pH 11.0 is that if great care is not taken the solution is very prone to hydrolyze at a high pH. The tendency for hydrolysis is a function of time and temperature as well as pH. If held at pH 11.0 at 180°F., hydrolysis will start in 30 min., but when held at 80°F. and pH 11.0 there will be no hydrolysis in 24 hr.

Conversely even at 180°F. the tendency for hydrolysis is greatly reduced if the solution is cooked at a pH of only 9.0. In the paper industry, solutions are usually cooked at 130°F. while in the paint and adhesive field solutions are cooked at 170°F. This is because paper coaters are afraid of alkaline hydrolysis with a loss in adhesive strength and are not interested in long term stability. Adhesive manufacturers know

Table XI. Effect of Solids on Viscosity of Casein Coating with 15% Adhesive Ratio

	% Solids 45.0	42.5	40.0	37.5	35.0
	As Made				
Viscosity, cp.—25°C.					
10 r.p.m.	2000	1350	320	100	40
20 r.p.m.	1850	1275	320	90	50
50 r.p.m.	1420	920	264	100	64
100 r.p.m.	1070	760	234	120	84
pH	9.6	9.6	9.6	9.6	9.6
	1 Day				
Viscosity, cp.—25°C.					
10 r.p.m.	2900	620	200	100	40
20 r.p.m.	2300	550	220	110	40
50 r.p.m.	1600	452	208	112	64
100 r.p.m.	1150	360	194	128	82
pH	9.5	9.6	9.6	9.6	9.5
Critical wax pick	9	9	9	9	9
IGT Values No. 4 ink—50 kg.—spring drive A, cm./sec.	132	105	138	105	155

Table XII. The Effect on the pH and Viscosity of a 15% Solid Casein Solution and 45% Solid Coating When Using Various Amounts of Ammonium Hydroxide

	A	*B*	*C*	*D*	*E*	*F*
NH_4OH on casein, %	4	6	8	10	12	14
15% Solid casein solution						
Viscosity, cp.—25°C.						
50 r.p.m.	1220	1240	1180	1620	1200	1440
pH	6.9	8.2	8.9	9.2	9.3	9.4
45% Solid casein coating						
Viscosity cp.—25°C.						
10 r.p.m.	7800	3600	2400	2400	1900	2200
20 r.p.m.	5800	3200	2250	2150	1800	2000
50 r.p.m.	3520	2160	1640	1560	1360	1480
100 r.p.m.	2320	1580	1160	1140	1000	1070
pH	7.7	8.5	9.1	9.4	9.6	9.7
Critical wax pick	9	9	9	9	9	9
IGT pick—no. 4 ink—50 kg.—spring A, cm./sec.	119	144	126	81	71	71

that if solutions are not cooked at 170°F. they will thin out on standing 1 month even if protected against spoilage. We feel this is because of the presence of enzymes that cannot be readily destroyed chemically but must be destroyed by cooking at about 170°F. The effect of cooking temperature on the viscosity of an ammonium hydroxide cut casein solution with pH 9.5 at 15% solids with 0.5% Dowicide A+G is shown in Table XIII. The solutions were all cooked 20 min. at the time shown. The data indicate that initial cooking temperature has little effect on viscosity as made but is quite critical if long term stability is desired. The effect of cooking time at temperatures of 140, 160, and 180°F. was also studied. When cooked 10 to 80 min. at 140°F. the initial viscosity was the same, and all thinned out on aging. At 160°F. all had the same initial viscosity but those cooked 40 min. or less thinned out on aging but one cooked 80 min. did not thin out. When cooked at 180°F. with 40 or 80 min. cooking there was some loss of viscosity as made. On aging none of these thinned out.

Effect of Type Mixer

The next variable to be studied was the type mixer used to prepare the coating. Coatings were prepared using six different laboratory mixers, the results of this work are shown in Table XIV. While high speed stirrers such as the Cowles or Hamilton-Beach do an excellent job of mixing they whip in considerable foam. The high viscosity of coating A and B was due to entrapped air. This did not leave on standing overnight. Most of this foam was of the coarse type and could probably have readily been eliminated with a defoamer. The results obtained with the other four stirrers was relatively uniform. This indicates that if air is not whipped into the coating the type stirrer is not too important. The exception was coating E made with a slow speed Hobart paddle mixer. In this case poor mixing was obtained and the coating had about 1 point lower wax pick than the others. Coatings A and B had about 1 point higher pick but this was probably because they were very foamy. In Table III it is noted that company C used a Hamilton-Beach mixer. They in turn had a higher viscosity than the other laboratories and 1

Table XIII. The Effect of Cooking Temperature on the Viscosity of a 15% Solid Casein Solution

Cooking temperature °F.	*120*	*140*	*160*	*180*
Viscosity, cp.—50 r.p.m.—80°F.				
As made	10,300	10,400	11,000	10,000
1 Week	4,300	6,300	12,000	9,900
1 Month	448	1,640	8,800	12,000
3 Months	68	252	10,200	10,300
6 Months	36	60	8,300	14,400

Table XIV. Effect of Type Mixer on Viscosity of Casein Coating at 45% Solids with 15% Adhesive Ratio

	Type Mixer					
	A	*B*	*C*	*D*	*E*	*F*
	Cowles dissolver	*Hamilton-Beach*	*Waring Blendor*	*Variable speed elec. stirrer*	*Hobart mixer*	*Heavy duty air stirrer*
As Made						
Viscosity, cp.—25°C.						
10 r.p.m.	9000	4600	2300	1900	2100	1900
20 r.p.m.	6400	3250	1900	1550	1900	1800
50 r.p.m.	4160	2060	1320	1080	1380	1360
100 r.p.m.	2860	1520	1010	770	990	1000
pH	9.3	9.3	9.4	9.2	9.4	9.3
24 Hours						
Viscosity, cp.—25°C.						
10 r.p.m.	9200	6700	2100	2200	2100	2000
20 r.p.m.	6100	4450	1750	1850	1850	1650
50 r.p.m.	3760	2740	1280	1280	1300	1160
100 r.p.m.	2440	1850	880	885	950	860
r.p.m.	9.1	9.2	9.3	9.1	9.1	9.3

point higher wax pick. This was probably due to the foam present.

Effect of Defoamer

Since the presence of foam was found to influence both the viscosity and adhesive strength of the coating it was felt worth while to study the effect of a defoamer. For this work we chose tributyl phosphate (TBP) rather than a prepared defoamer. The results of this work are shown in Table XV. Coating A was the control made under standard conditions. In coating B, 2 drops of TBP were added to the clay slurry before adding the casein solution. The viscosity was somewhat lower but coating A had only little foam to start with.

In coating C, 2 drops of TBP were added to the casein solution after cooking. It also lowered viscosity slightly.

In coating D a second casein solution (2) was used, and in this test no defoamer was present. In coating E, 2 drops of TBP were added to the clay slurry. In this case it seemed of little help.

In coating F, defoamer was added to the casein before cooking and also to the final coating. Here it showed little effect.

In coating G, defoamer was added to only the casein before cooking. It again showed little effect.

The addition of defoamer to the casein solution E versus F reduced viscosity slightly.

In conclusion if care is taken in mixing the coating no defoamer is required, but if air is whipped in during mixing a

Table XV. Effect of Defoamer on Viscosity of Casein Coating at 45% Solids

	Code						
	A	*B*	*C*	*D*	*E*	*F*	*G*
Casein solution no.	1	1	1	2	2	3	3
Defoamer in solution	No	No	No	No	No	Yes	Yes
Viscosity cp.—25°C. 50 r.p.m.	1640	1640	1640	1720	1720	1520	1520
Viscosity of coatings, cp.—25°C.							
10 r.p.m.	3100	1800	2200	1900	1600	1700	1800
20 r.p.m.	2450	1550	1900	1650	1500	1600	1600
50 r.p.m.	1660	1120	1380	1200	1120	1240	1220
100 r.p.m.	1170	820	1000	870	840	920	900
Defoamer added	No	Yes	Yes	No	Yes	Yes	No

A—No defoamer in casein solution or coating.
B—No defoamer in casein, 2 drops of TBP in clay slurry.
C—No defoamer in casein as cooked but 2 drops of TBP added after cooking.
D—No defoamer in casein solution or coating.
E—No defoamer in casein solution, 2 drops of TBP in clay slurry.
F—2 drops of TBP added to casein before cooking and 2 drops added to final coating.
G—2 drops of TBP added to casein before cooking.

Table XVI. Effect of Type Casein and Alkali on Viscosity of 15% Solid Casein Solution and 45% Solid Casein Coating

	Code 1	2	3	4	5	6	7	8	9	10	11
Type Casein	A	B	C	D	E	F	G	A	A	A	A
Type alkali	NH_4OH	NH_4OH	NH_4OH	NH_4OH	NH_4OH	NH_4OH	NH_4OH	Borax	NH_4OH + Borax	N_aOH	N_aOH
15% Solid casein solution											
Viscosity, cp.—25°C.											
50 r.p.m.	1640	1720	680	1580	1200	316	40	1540	2480	1120	1280
pH	9.4	9.4	9.2	9.2	9.4	9.4	9.4	9.0	8.7	6.4	8.3
45% Solid coating—15% adhesive ratio											
Viscosity, cp.—25°C.											
10 r.p.m.	3100	2800	1300	2500	1200	1200	780	..	2800	..	3700
20 r.p.m.	2450	2400	1100	2150	1150	1025	500	..	2550	..	3300
50 r.p.m.	1660	1680	780	1400	900	750	280	..	1760	..	2200
100 r.p.m.	1170	1220	570	1000	690	585	188	..	1250	..	1550
pH	9.3	9.3	9.1	9.0	9.3	9.4	8.8	..	9.2	..	9.3
Critical wax pick	9	9	9	9	9	9	4	..	9	..	9

small amount of defoamer should be added to the casein solution.

Effect of Type Casein and Alkali

Using the standard procedure, solutions of seven different type caseins were prepared. These tests included some highly modified caseins as well as regular lactic and hydrochloric caseins.

Casein A was the lactic casein used throughout the series. Caseins A, B, and D had about the same viscosity in the solution and coating. Casein C had less than half the viscosity of casein A and gave a coating with half the viscosity of coating 1. Casein E was slightly lower in viscosity than casein C and gave coating 5 with slightly lower viscosity. Casein F had half the viscosity of casein C but gave coating 6 with viscosity equal to coating 3. In all of this work no significant difference in adhesive strength could be found between coating 1 through 6. Casein G had $^1/_{40}$ the viscosity of casein A and gave coating 7 with $^1/_{10}$ the viscosity of coating 1. In this case a sharp loss in adhesive strength did occur.

In coatings 8 through 11 the same casein was used but the alkali was varied. With coatings 8 and 10 severe flocculation was obtained due to the low pH of the casein solution. For coating 8 the casein was cut with 15% borax, and for coating 10 the casein was cut with 1.8% NaOH. When a combination of borax and ammonium hydroxide were used as a coating 9 there was little effect on the viscosity of the coating but the viscosity of the casein solution increased by one-third. In coating 11 the amount of NaOH was increased to 3.0%. The solution had a lower viscosity than in coating 1 but coating 11 was 20% higher in viscosity than coating 1.

The results show that similar type caseins give approximately the same viscosity in solutions and coatings. Modified caseins vary considerably in viscosity. It is also seen that the viscosity of the casein solution does not always correlate with the viscosity of the coating.

Adhesive Ratio

In Table XVII the effect of adhesive ratio on viscosity and

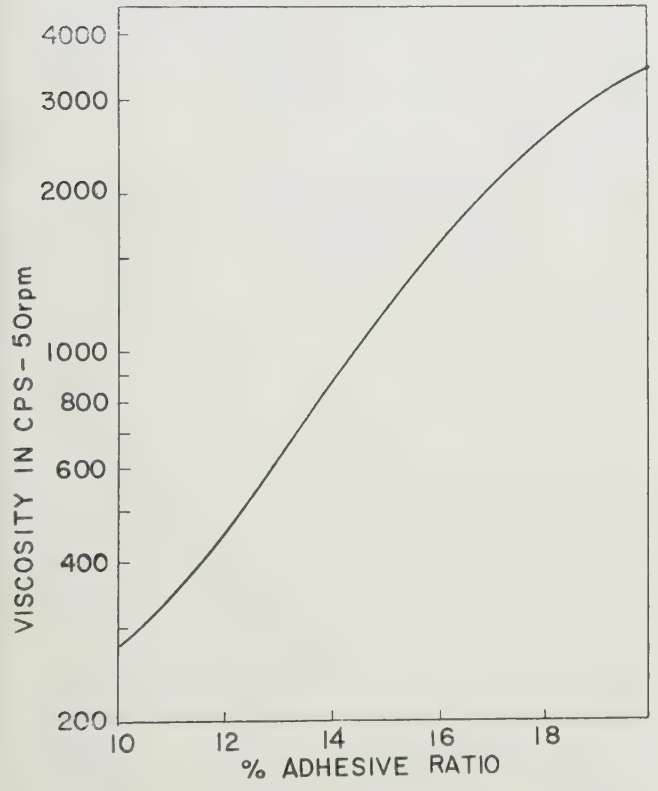

Fig. 4. Effect of adhesive ratio on the viscosity of a casein coating at 45% solids

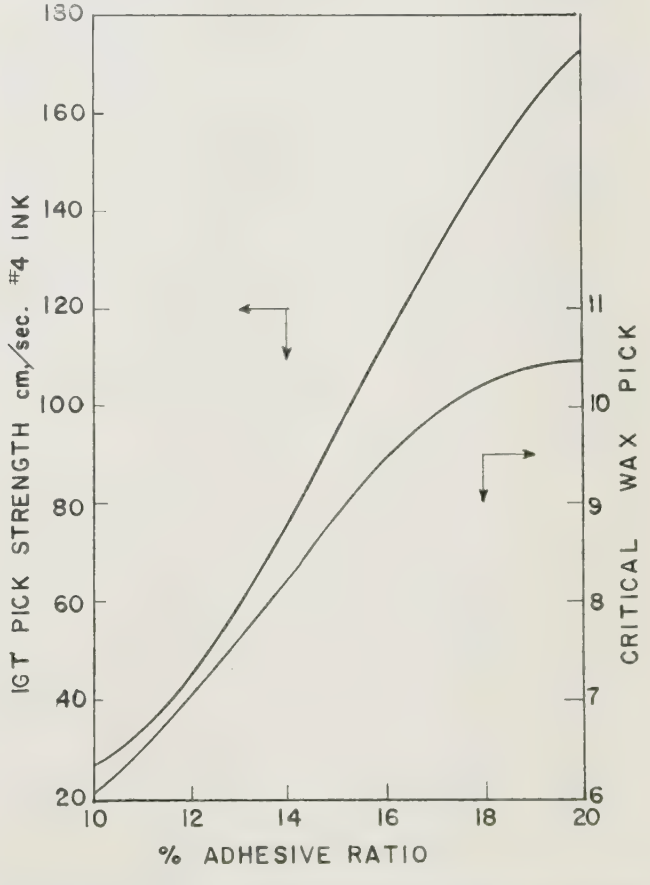

Fig. 5. The effect of adhesive ratio on the wax pick and IGT pick strength of a 45% solid casein coating

Table XVII. Effect of Adhesive Ratio on Viscosity and Strength of Casein Coating at 45% Solids

	Adhesive ratio, %								
	20	18	16	15	14	12	12	10	10
Defoamer Added									
Viscosity, cp.—25°C.									
10 r.p.m.	7500	4800	3000	2000	1700	1300	850	1100	350
20 r.p.m.	5500	3500	2300	1700	1500	1025	675	900	325
50 r.p.m.	3500	2320	1600	1220	1040	680	500	700	280
100 r.p.m.	2550	1700	1140	900	770	545	420	590	245
pH	9.3	9.4	9.4	9.5	9.5	9.5	..	9.5	..
Critical wax pick	10.5	10	9.5	9.0	8.0	8	7	7	6
IGT value no. 4 ink—50 kg.—spring drive A, cm./sec.	173	155	98	90	73	79	52	105	25

adhesive strength is shown. For the coatings at a 20 and 18% adhesive ratio the casein solution had to be prepared at 17% solids. In coatings with an adhesive ratio of 12% and 10% considerable foam was encountered. It was therefore necessary to add 2 drops of TBP to the final coating. It can be seen that this reduced viscosity considerably. When the coating was foamy it had a 1 point higher wax pick rating. In this case the importance of defoamer was evident. In Fig. 4 the effect of adhesive ratio on viscosity is shown.

In Fig. 5 the relationship between adhesive ratio and adhesive strength is shown using both the wax pick and IGT values. Based on the above relationship, in Fig. 6 we tried to show a relationship between critical wax pick and IGT pick strength using the spring drive at A speed and 50 kg. pressure with a no. 4 ink.

Sufficient data were not available to plot this curve with great accuracy. The points were very scattered, and the figure is only intended to show the general relationship between the two values. When a wax pick of 10 or higher was obtained the IGT value was always high and when the wax pick was 8 or lower the IGT was always low. When the wax pick was 9, there were many IGT values both high and low.

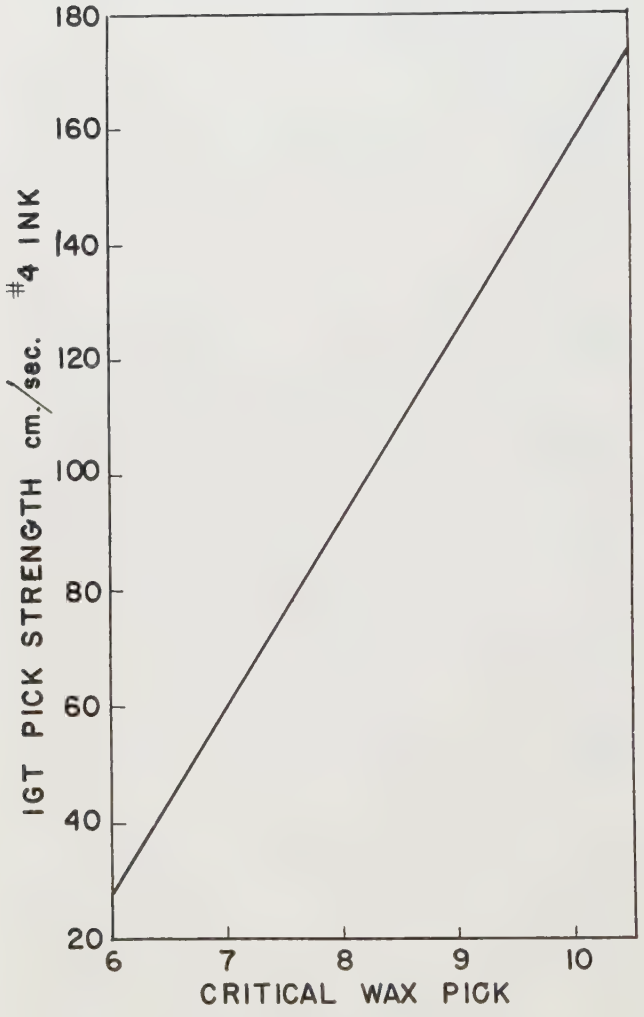

Fig. 6. The relationship of wax pick to IGT pick strength

Effect of Latex

In practice today, casein is seldom used as the sole adhesive in a paper coating. Usually from 20 to 80% of the casein is replaced with a styrene-butadiene or acrylic type emulsion. This is done because the latex reduces viscosity, improves flexibility, improves gloss and smoothness on calendering, and gives better varnish holdout. In Table XVIII, the effect of replacing a portion or all of the casein with styrene-butadiene (Polyco 2419) or acrylic (Polyco 2712) is shown. By replacing 20% of the casein with latex a slight reduction in viscosity is obtained, but when using 40% or more latex a considerable reduction is obtained.

A very significant fact is the relationship between the wax pick values and the IGT values. It is generally recognized that when latex is present the wax pick values read low. However, previous to these data few figures have been published on this subject. The results show that while the wax pick values are lower when latex is present the IGT values increase. This is particularly evident in coating 9 which shows no picking at 205 cm. per sec. on the IGT with a wax pick of 6 compared to coating 1 with a wax pick of 9 and IGT value of 164 cm. per sec.

The general improvement in adhesive strength with calendering should also be noted.

FACTORS EFFECTING ADHESIVE STRENGTH

Some of the factors effecting adhesive strength have already been discussed, but still other variables were also studied not related to the coating.

Drawdowns were made on both sides of the standard paper and also sideways. This did not affect adhesive strength. The coatings were airdried and dried at various times and temperatures. None of these factors changed adhesive strength significantly. Drawdowns were laid flat and hung in the oven, but this did not affect adhesive strength. Drawdowns were made on glass and a metal vacuum suction plate. This did not affect adhesive strength. Drawdowns were made using the 0.0015, 0.005, and 0.006-in. Bird film applicators. Coat weights of 26.1, 41.6, and 97.0 lb. per ream (25 × 38—500) were applied. The wax pick values were 9, 10, and 11 on these three drawdowns. This indicates that as coat weight is increased adhesive strength increases. The average coat weight applied by the 12 laboratories using an 0.001-in. Bird film applicator was 16.8 lb. per ream.

Drawdowns of the standard coating were made on unbleached kraft paper, bleached kraft board, and newsbacked cylinder board. In all cases a wax pick value of 9 was obtained.

One of the greatest variables encountered was the taking of

Table XVIII. Effect of Various Amounts and Types of Latex on the Viscosity and Adhesive Strength of a Pigmented Casein Coating at 45% Solids and 15% Adhesive Ratio

Type latex →	Code: A	B	C	D	E	F	G	H	I
		Styrene-butadiene				Acrylic			
% Latex on adhesive, % →	0	20	40	80	100	20	40	80	100
Viscosity, cp. 25°C.									
10 r.p.m.	1900	550	60	40	20	850	80	80	20
20 r.p.m.	1800	575	60	30	30	825	80	60	20
50 r.p.m.	1360	520	72	40	28	670	84	68	28
100 r.p.m.	1000	470	98	50	32	560	106	94	32
pH	9.6	9.4	9.5	9.5	9.7	9.2	9.2	8.4	7.5
Critical wax pick									
Uncalendered	8	7	6	5	5	7	6	6	5
Calendered	9	8	6	6	7	7	7	7	6
IGT Pick no. 4 ink—50 kg.—spring A									
Uncalendered, cm./sec.	90	71	38	105	120	52	71	159	183
Calendered, cm./sec.	164	120	106	144	120	133	133	175	205

the wax picks themselves and determining the end point. For a study of this variable we used a control sheet of a commercially coated, all casein, offset paper obtained from the Newton Falls Paper Co.

It was found that if the wax was pulled off before allowing it to cool, the wax pick could vary by one point from 10 to 9. However, no matter what was done pick was never obtained at 8 but always with 11. After 4 min. cooling the results were quite constant.

After heating the wax, intervals of 1, 2, 4, 8, and 16 sec. were allowed before applying the wax. A 1 to 4 sec. delay gave little change in adhesive strength but if over 8 sec. was allowed, the wax was too cool to adhere under any conditions.

When using old or new waxes, long or short ones, we found only minor variations in adhesive strength.

The pressure used when applying the wax did not seem to have much effect on the results.

The force or speed when removing the wax varied the results by one wax pick. When the 10 wax was pulled off with a quick jerk no pick was obtained, but when it was pulled off slowly, pick was obtained.

In many cases we found that out of four tests two might show no pick with a 10 wax and two slight pick with a 10 wax. We have not been able to find whether this is due to the coating, paper, wax, or test procedure.

A serious problem encountered was the determination of the end point. Some laboratories called it picking if the slightest edge cracked, while others felt a significant degree of picking was required. In defining the end point it might be well to always use the terms suggested by Dennison, that is indicate no pick 9, slight edge pick 10, considerable edge pick at 11, body stock pick at 12.

SUMMARY

1. The proposed method for preparing a 15% casein solution for viscosity tests does not give good reproducible results. By the last series of cooperative viscosity tests an average viscosity of 1401 cp. with a standard deviation of 539 cp. was obtained.

2. The method for preparing a pigmented coating at 45% solids seems to give satisfactory coating viscosity tests. At 50 r.p.m. a viscosity of 950 cp. with a standard deviation of 132 cp. was obtained.

3. Using the suggested procedure for making drawdowns, a Dennison wax pick value reproducible to ±0.5 units can be obtained.

4. Further work seems necessary on the IGT Test.

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The authors wish to thank all members of ASTM Committee D-25 Subcommittee IV who participated in these tests. In addition we wish to acknowledge the help and cooperation of the Georgia Kaolin Co. for supplying the clay slurries and the Newton Falls Paper Co. for supplying the raw stock used in these tests.

The Economics of Producing Circular Shapes

JOSEPH G. DEBO

A formula is developed whereby the number of circles which may be cut from a given area most economically can be predicted.

Predicting the number of square yards required to produce a given number of circles is not always as straightforward as it might seem. Where numerous diameters and widths of material are involved, a physical layout on the drawing board becomes impractical and is often deceiving. Impractical from the standpoint of number of combinations, deceiving because what looks like the best layout for a given set of conditions may not be the best from the standpoint of material usage. To illustrate, let us take the following example:

It is desired to produce 3-in. diam. circles. What would be the best layout to use?

The best use of material, cutting one at a time in a straight line, and neglecting trim and any spacing between adjacent cuts, would be about 78.6%. This same percentage holds true if two or three or more are cut at one time and succeeding cuts are made in a straight line (see Fig. 1).

The best use of material should come from such a die pattern as shown in Figs. 2 to 4, since the closest one can cut circles is so that lines between adjacent centers will form equilateral triangles.

Now let's compare requirements by both methods. In the first method (rectangular pattern), the area required for 16 3-in. diam. circles would be 144 sq. in., for 32 of 3-in. diam. 288 sq. in. and 432 sq. in. for 48 of 3-in. diam.

Switching to the second pattern (triangular), the width may be figured as follows:

$$w = d + (N - 1)d \sin A$$

where

w = width of material

d = diameter of circle

N = number of circles across material, i.e. (2) $N = 2$, (3) $N = 3$, (4) $N = 4$

A = angle between side of material and line between two adjacent centers across the material (2) $A = 60°$, (3) $A = 60°$, (4) $A = 60°$

The widths then become

$$w_{(2)} = 3 + (2 - 1)(3)\,0.866 = 5.596 \text{ in.}$$

$$w_{(3)} = 3 + (3 - 1)(3)\,0.866 = 8.196 \text{ in.}$$

$$w_{(4)} = 3 + (4 - 1)(3)\,0.866 = 10.794 \text{ in.}$$

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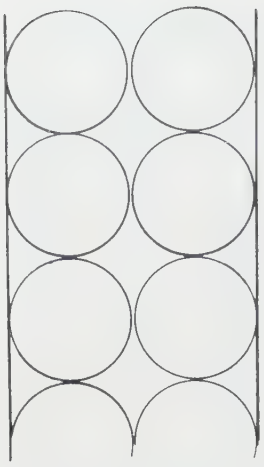

Fig. 1.

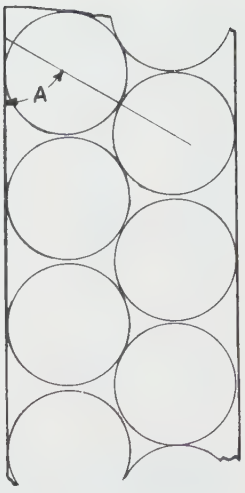

Fig. 2. N = 2

The lengths and corresponding areas may be figured as follows:

16 Circles

(2) $\frac{16d}{2} + \frac{d}{2} = 8d + 1^1/_2 =$
$25^1/_2$ in. × 5.598 in. = 142.749 sq. in.

(3) $\frac{16d}{3} + \frac{d}{2} = 5^1/_3 d + 1^1/_2 =$
$17^1/_2$ in. × 8.196 in. = 143.430 sq. in.

(4) $\frac{16d}{4} + \frac{d}{2} = 4d + 1^1/_2 =$
$13^1/_2$ in. × 10.794 in. = 145.719 sq. in.

32 Circles

(2) $\frac{32d}{2} + \frac{d}{2} = 16d + 1^1/_2 =$
$49^1/_2$ in. × 5.598 in. = 277.101 sq. in.

(3) $\frac{32d}{3} + \frac{d}{2} = 10^2/_3 d + 1^1/_2 =$
$33^1/_2$ in. × 8.196 in. = 274.566 sq. in.

(4) $\frac{32d}{4} + \frac{d}{2} = 8d + 1^1/_2 =$
$25^1/_2$ in. × 10.794 in. = 275.247 sq. in.

48 Circles

(2) $\frac{48d}{2} + \frac{d}{2} = 24d + 1^1/_2 =$
$75^1/_2$ in. × 5.598 in. = 422.649 sq. in.

(3) $\frac{48d}{3} + \frac{d}{2} = 16d + 1^1/_2 =$
$49^1/_2$ in. × 8.196 in. = 405.702 sq. in.

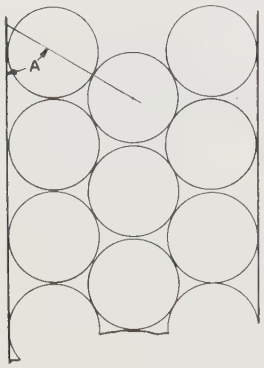

Fig. 3. N = 3

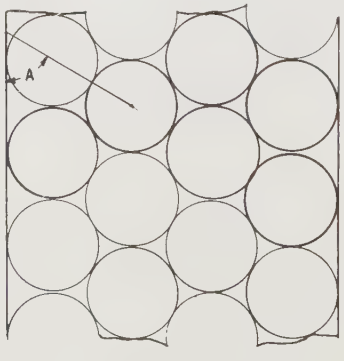

Fig. 4. N = 4

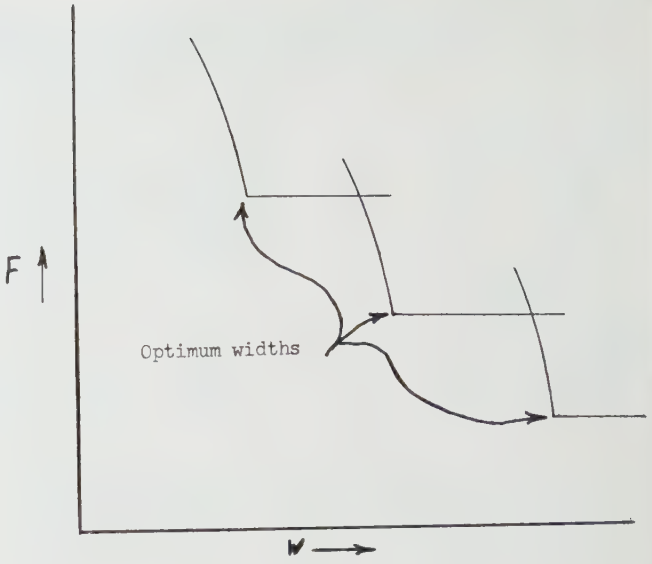

Fig. 5.

(4) $\frac{48d}{4} + \frac{d}{2} = 12d + 1^1/_2 =$
$37^1/_2$ in. × 10.794 in. = 404.775 sq. in.

It will be noted that as the quantity increases, the difference between the area requirements using 2, 3, and 4 circles in width becomes smaller. This continues until a quantity is reached where less area is required for the same production by cutting more across the width.

This then means that all things being equal, the wider the material being cut into circles, the more economical is the use of that material.

The question may well be asked then, "If the material width available does not coincide with the optimum width of the diameter desired, is it better to squeeze another circle in or trim back to the next smaller optimum width?"

Since many variables enter into such a decision, the best that can be done is to explain what happens.

The variations possible from the previously discussed conditions are basically two in number if the diameter is constant. There are:

1. The material is wider than the optimum pattern. Here a margin of uncut material will remain if the same pattern of cutting is used. However, spreading the pattern out shows no improvement in use of material until another circle can be cut from the width, and succeeding cuts must be made the same distance apart in spite of the flattened pattern.

2. The material is narrower than the optimum pattern. Here the pattern must be compressed sideways if the same number of circles are to be cut across the width. This pattern, however, requires an increase in indexing over the optimum pattern.

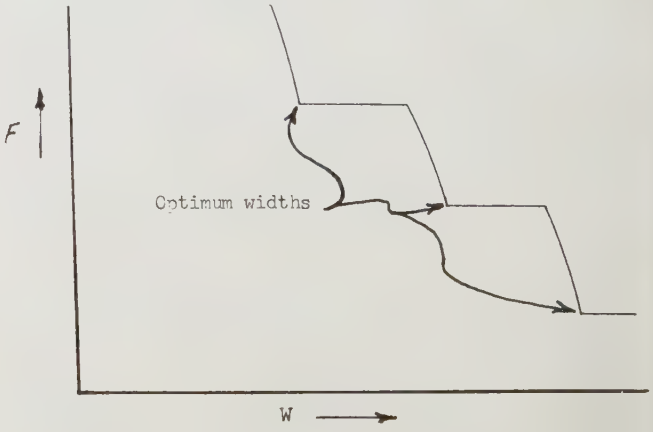

Fig. 6.

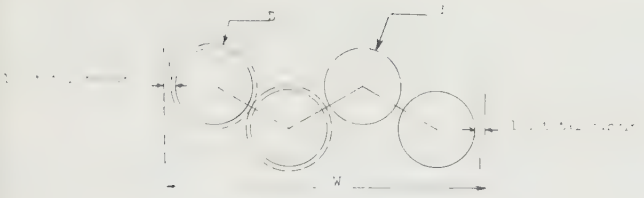

Fig. 7.

To determine where one should add or subtract a circle, one must compute the indexing required under given conditions of width. This can be done by solving:

$$\text{Index} = 2d \text{ cosine } A$$

A being derived from the previous formula

$$w = d + (N - 1)d \text{ sine } A$$

Since the index must be at least equal to d then A must $\leqq 60°$. Determining a factor as follows:

$$F = \frac{\text{Index}}{36N} = \text{Linear yards per circle}$$

By plotting this F against w on each side of the optimum widths, one obtains a series of curves for each diameter considered, as in Fig. 5.

Simplified, each curve would look like Fig. 6.

This gives the lowest value of F for a given diameter and width of material. Multiplying this F by the number of circles desired gives the required number of yards of material w in. wide.

Consideration can be given to the spacing required between circles for most cutting operations by letting the d in the formula equal the diameter plus the spacing.

The margin on the sides of the material can be added as a separate quantity minus the spacing between two adjacent circles.

The final formula for width then becomes:

$$w = (\text{total margin-spacing}) + D + (N-1)D \text{ sine } A$$

where D = circle diameter + spacing between adjacent circles.

This formula is illustrated by Fig. 7.

The advantage of this formula is the consideration given to all the variables involved in pattern changes. With it, one may anticipate the economics of material usage versus the costs of additional slitting.

Received July 5, 1961.

The Application of High Yield Pulping Techniques to Hardwoods and Softwoods for the Production of Insulation and Hardboard Stocks

LEE EBERHARDT

This paper discusses the application of high yield pulping techniques, i.e., groundwood from chips, cold soda, N.S.S.C., steamed wood and hot sulfite impregnation, to hardwoods and coniferous woods for the production of insulation board stock. The various processes, the resultant pulp characteristics, and production equipment are detailed. Results of semicommercial trials applying high yield processes to a number of conventionally used hardwoods and softwoods are presented. This work indicates many of these pulps while marginal in board making quality when used alone, may be blended into composite furnishes to produce superior boards.

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At the 44th Annual Meeting two years ago a paper titled "Latest Trends in Raw Materials and Process in the Insulation and Hardboard Fields" was presented. At that time, it was indicated that future trends in wet end processing would involve wider application of high yield processes to a wider variety of wood species for the production of insulation and hardboard machine furnishes. The interim period has seen considerable research, semicommercial pilot plant work, and commercial application of high yield pulping techniques in the board industry.

This paper covers some of this activity and will attempt to summarize and provide an up-to-date picture on the possibilities of the various types of high yield pulps. Most of the data is based upon semicommercial pilot plant studies at Fiber Products Laboratory. The balance of the data comes from commercial operations:

HIGH YIELD PROCESSES SUITABLE FOR INSULATION BOARD

The following high yield processes are being applied experimentally and commercially for the production of insulation boards. They are listed according to yield based on wood in descending order: (1) groundwood, (2) steamed wood, (3) cold soda, (4) sulfite impregnated wood, and (5) neutral sulfite semichemical. The first two processes have, of course, been used for the production of insulation board for years. The pioneer manufacturing method being groundwood. Steamed wood arrived a little later, but is the basic method for production of hardboard pulps and is a major process for insulation board.

In general, each process involves use of heat, chemical, and power in varying degrees. The equipment utilized is nearly identical in each case varying only according to the variation in the factors of heat, chemical, and power. The exception being groundwood production which has historically been produced in stone grinders. However, introduction of refiner-groundwood or groundwood from chips removes the equipment differential. In fact, if the chips are from dry waste materials, water impregnation may be required. In which case, the systems become nearly identical.

Groundwood from Chips

Groundwood historically has been widely used as a major component for production of insulation board stock. The woods used have been the grindable species, i.e., coniferous woods and low density hardwoods such as poplar, aspen, cottonwood, and willow.

The pulps are characterized by light color, fair strength, and good density. Generally, they produce a brittle board with good stiffness, but poor bending quality. Handleability leaves something to be desired. Strength properties have been improved by extensive use of starch and/or wastepaper. Low freeness groundwood pulps have excellent blending properties when used with free stocks such as Asplund fiber steamed wood and hot sulfite impregnated pulps.

The stone grinder is handicapped due to relatively rigid raw material requirement, and limited range of pulp quality that can be produced by a single grinder.

Wood to the grinder must be 2 or 4 ft. straight pulpwood with diameter of blocks in the range of 6 to 16 in. To produce such material, yield is reduced by the grinder's inability to use waste, created all the way from the tree cut in the bush to the grinding unit. Obviously yield is improved by chipping. The use of chips from sawmill waste, a growing raw material source, cannot be utilized by grinders.

Pulp quality in the stone grinder can only be controlled by the stone specification, burring of stone, and pressure applied to the wood against the stone. Once the stone specification is established, this factor cannot be changed without stone replacement. Burring and pressure will only produce relatively minor changes in stock quality. This handicaps the mill

Table I. Southern Pine (*Pinus taeda* and *P. echinata*) and Oak (*Quercus minor* and *Q. rubra*) Processed by Cold Soda at Several Chemical Usage Levels to Produce Insulation Board Stock

Run no.	Type material	Chemical consumed, %	Approx. washed yield, %	Refining, hp.-days/ ADT	SFMC freeness, washed pulp, sec.	Board weight per M sq. ft. 1/2-in. basis	U.S. break load	Modulus of rupture	Remarks
IA	Pine	10	80	17.2	13.2	708	16.2	398	Yield too low. Excessive treatment
IB	Pine	6	85	26.1	28.4	683	21.8	512	Excellent board
ID	Pine	3.6	88	26.5	22.6	712	19.7	456	Excellent board
IC	Pine	5.3	86	20.3	15.0	706	16.5	396	Adequate board
IE	Oak	6.7	84	10.3	26.8	717	15.6	379	Good board
IF	Oak	6.3	84	12.9	48.0	733	12.4	290	Marginal board
IG	Oak	4.0	89	11.0	17.6	699	6.5	156	Quality inadequate. Additional refining would improve
IH	Composite board; 67% IC and 33% IF	Composite av. 5.6%	Composite av. 85%	Composite av. 16.8	18.8	700	14.0	336	Good board. Components produce excellent blend in spite marginal 2F

Note: 1. In all runs except ID & IG residual NaOH and pH was so high pH correction and sizing costs would be prohibitive without washing. 2. All boards were relatively dark particularly the oaks. 3. Prerefining treatment—cold soda, 10 min., impregnation at 150 p.s.i.

with a limited range of stock quality. Another difficulty is change in stock quality as the stone wears between burrings. These deficiencies and difficulties are partially overcome in multiple grinder installations where the overall tonnage is an average of the individual grinders. Producing uniform quality board in a small mill equipped with only one or two grinders on the other hand is nearly an impossibility.

The last two years has witnessed the perfection of the groundwood from chips using disk refiners. It has now been proved that the double disk refiner will produce equal or better quality pulp at equal power input when compared with a stone grinder. The range of pulp quality is greatly widened in the disk mill, since it is possible by merely changing refiner plate clearance to produce stocks with freenesses ranging from book grade groundwood to high freeness insulation board stock.

The system is relatively simple, in that the wood is chipped, run through an impact mill for producing uniform sizing and then screened for removal of fines. The chips are then metered to one or more double disk refiners in parallel (number required proportional to tonnage requirement) which in a single pass produces the stock quality required. The only screening required on the stock is of the coarse variety for removal of any chips that might by accident get into the system.

If sawmill waste or chips from outside sources are to be used, it is suggested the above system be supplemented with a chip washer for removal of tramp materials. Excellent units of this type are installed at the Pilot Rock, Ore., plant of United States Gypsum Co., and Longview Fibre Co., Longview, Wash.

If extremely dry materials are to be used such as kiln dried waste, veneer clippings, veneer cores, etc., it is possible to utilize a digester unit such as the Rapid Cycle, Grenco, or M & D units for restoring moisture back into the wood (hot water impregnation) or dilute caustic solutions may be used. The caustic acting as a wetting agent.

Groundwood from chips expands the possibilities of groundwood-type fiber for insulation board stock considerably. The economics are very much improved due primarily to increased yield, and use of lower cost raw materials. The method provides a very versatile system and can provide the first step toward the other high yield type pulps with their greater range of wood species utilization possibilities.

Steamed Wood

The first process developed for insulation board following groundwood was that of steamed wood in its several varieties such as Asplund fiber, gum stock, rapid cycle digester stock, etc. This has always been the primary fiber process for production of conventional hardboard stocks and in combination mills producing both insulation and hardboard.

Table II. Southern Gum (*Liquidambar styraciflua*) Processed by Four High Yield Methods to Produce Insulation Board Stock

Run no.	Prerefining treatment (process)	Refining, hp.-days/ ADT	SFMC freeness,[a] sec.	Board weight per M sq. ft. 1/2-in. basis	U.S. break load	Modulus of rupture	Board color[b]	Remarks
2A	None (groundwood from chips)	16.1	8	643	1.1	27	Fair. Depends on percent heartwood	Very free—low power and strength. Increasing power would not give adequate test
2B	Steamed 10 min. at 50 p.s.i. (steamed wood)	28.8	16	700	5.8	139	Darker than IA	Too weak—could be improved but doubtful if enough
2C	Steamed 10 min. at 150 p.s.i. (steamed wood)	11.7	34	707	18.4	442	Very dark	Overcooked—commercial quality—could be improved by less cooking
2D	Impregnate hot Na_2SO_3 (280°F.) 10 min. at 100 p.s.i. (hot sulfite)	20.9	12	738	11.0	260	Lighter than IA	Nearly commercial quality—good blending pulp
2E	Impregnate cold NaOH 20 min. at 150 p.s.i. (cold soda)	12.3	26	744	20.7	496	Darker than IA	Good commercial quality—power may be abnormally low

[a] SFMC freeness taken on washed unsized stock 2D and 2E, and unwashed, unsized stock for 2A through 2C.
[b] For color reference stock 2A is approximately 50 to 52% G.E. brightness and 1C approximately 35% G.E. brightness.

Table III. Southern Pine (*Pinus echinata*) and Oak (*Quercus Marylandica*) Processed by Several High Yield Techniques for the Production of Insulation Board

Run no.	*Type material*	*Prerefining treatment (process)*	*% Chem. used*	*Approx. yield washed, %*	*Refining, hp.-days/ ADT*	*SFMC freeness, sec.[a]*	*Board weight per M sq. ft. 1/2-in. basis*	*U.S. break load*	*Modulus of rupture*	*Remarks*
3A	Pine	Steamed 4 min. at 175 p.s.i. (steamed wood)	None	90	17.2	16.4	726	10.7	271	Overcooked—stock too soft —dark
3B	Pine	Steamed 3 min. at 150 p.s.i. (steamed wood)	None	93	22.3	19.2	683	15.5	373	Excellent board—dark
3C	Pine	None (groundwood from chips)	None	95	16.1	14.3	592	8.1	118	Needs more power—board too weak and light
3D	Pine	Impregnate hot (280° F.) Na_2SO_3 at 100 p.s.i. 10 min. (hot sulfite)	3.5	88	26.5	17.0	550	12.4	298	Extremely light weight—light color
3E	Pine	Same as 3D except cooked 4 min. at 150 p.s.i. (NSSC)	3.5	85	20.4	12.0	528	8.8	170	Extreme light weight—light color
3F	Pine	Impregnated cold NaOH 150 p.s.i. 10 min. (cold soda)	4.0	88	24.3	20.0	706	17.5	414	Good board—fair color (yellowish)
3G	Oak	Steamed 4 min. at 140 p.s.i. (steamed wood)	None	92	15.5	63.0	732	13.6	305	Adequate board—dark
3H	Oak	NaOH impregnated 100 p.s.i.—10 min. (cold soda)	3	90.2	9.9	41	658	9.9	237	Inadequate strength—very dark
3J	Oak	Same as 3H. Cold soda	3	90.2	17.8	112.	Too slow to form board			Good blending stock
3K	80% Oak 20% Pine	Steamed 4 min. at 140 p.s.i. (steamed wood)	None	91.5	16.5	16.9	802	11 7	296	Marginal
Comp 1	50% Pine 50% Pine	Steamed wood 3A Hot sulfite 3D	1.8	89	21.9	17.0	885	26.7	642	Heavy but exceptional strength—good color
Comp 2	50% Oak 50% Pine	Cold soda 3J Hot sulfite 3D	3.25	89	22.2	32	718	17.1	410	Excellent in all respects
Comp 3	25% Pine 75% Oak	Hot sulfite 3D Cold soda 3J	3.1	89	20.0	34	738	29.3	674	Excellent in all respects
Comp 4	60% Pine 40% Oak	Steamed wood 3B Cold soda 3J	0.4	92	20.3	27.4	804	13.0	312	Adequate—dark color
Comp 5	60% Pine 40% Oak	Steamed wood 3B Steamed wood 3G	None	93	19.9	24.3	767	12.4	305	Marginal—dark

[a] SFMC freeness on chemically treated pulps on washed unsized stock. SFMC freeness on groundwood from chips and steamed wood on unwashed, unsized stock.

Steaming wood provides a stronger more flexible fiber than groundwood at the handicap of darkening the board depending upon the severity of the treatment. Yields are a little lower than groundwood, and refining power requirement is lower, about offsetting the cost of the steam used.

Following the steaming, the chips are refined to pulp using disk mills. The double disk is particularly effective and widely used, the single disk unit has proved satisfactory in some cases.

After refining in many cases, the stock is washed to remove water solubles. Washing is almost mandatory in hardboard (conventional) stocks to reduce sticking of boards on press caul plates and platens.

Cold Soda Pulping

The Forest Products Laboratory, Madison, Wis., developed (experimentally) the cold soda process a number of years ago. This process used on hardwoods primarily of the low density type initially produced excellent high yield papermaking pulps suitable for inclusion in newsprint and book papers. Semicommercial pilot plant work was undertaken at Bauer's Fiber Products Lab in Springfield, and resulted in a number of installations in this country and abroad producing a wide variety of paper type pulps. After the initial work, the process was extended to cover practically all the hardwood species. This work in the paper pulp field led to an interest in the board field, and it appears the pulps have excellent possibilities for insulation board, particularly as slow filler-type stocks.

The cold soda process consists of impregnation of the chips with hydrostatic pressure or atmospheric steeping for an extended period followed by refining in disk mills and subsequently screening, cleaning, and bleaching for paper pulps. The latter steps are generally omitted for board.

Impregnation can be handled by the rapid cycle digester or M & D type unit, and recently at Fiber Products Lab and in commercial installations, the Impressafiner has proved very effective. Refining is the same as groundwood from chips and steamed wood except power requirement for pulping lies between the two types.

As indicated previously, the cold soda process was developed primarily on hardwoods. It has recently been extended to the coniferous species for insulation board stocks. The method allows production of good quality stocks from dense hardwoods unadapted to groundwood methods.

Pulps produced are similar to groundwood except have generally improved strength properties. The cold soda treatment produces a darkening of the pulp of approximately 3 to 5% G.E., from the brightness characteristic of the species. This darkening is greatly accelerated by steaming or other heat application. This effect is accompanied by a loss in fiber strength and generally is not recommended. On some wood species, cold soda pulps take a yellowish cast which may cause difficulty in coating colors or produce a board with a poor unpleasing surface color.

Unfortunately, many wood species are naturally dark in color and the additional color drop due to cold soda processing may preclude their use in decorative type products.

Caustic application is generally less than 5% based on moisture-free wood weight yields are usually 85% or above again based on moisture-free wood. One difficulty that arises with the cold soda method is high pH of the pulp and residual caustic. In most cases, the pulp must be washed or difficulty in sizing will result. If pH control is by use of neutralizing acid or alum, costs become uneconomical. The best approach appears to be use of the minimum amount of caustic applied to the wood to produce the desired end product pulp, or extracting the surplus liquor from the chips with the

Table IV. General Summary of Board Making Qualities of Different Types Raw Material and Processes

	Process & raw material	Chemical	Approx. yield, %	Refining power	Strength	Bulk	Color	Remarks
4A	Groundwood from chips—coniferous woods	None	95	High	Fair	Fair	Good	Good board pulp in spite of low strength and brittleness
4B	Groundwood from chips—low specific gravity hardwoods	None	95	High	Fair	Fair	Depends on wood color	Not quite as good as 48
4C	Groundwood from chips—dense hardwoods	None	95	High	Poor	Poor	"	Not recommended
4D	Steamed wood—coniferous woods	None	90 to 95	Average	Good	Fair	Poor	Excellent board quality—color a problem
4E	Steamed wood—hardwoods	None	90 to 95	Average	Fair	Poor	Poor	Not quite as good as 4E but generally adequate—good blender
4F	Cold soda—softwoods	3 to 5% NaOH	85 to 90	Average	Excellent	Good	Fair	Excellent board quality
4G	Cold soda—hardwoods	3 to 5% NaOH	85 to 90	Average	Good	Fair	Poor	Good board quality—color a problem—excellent blender with 4A, 4H
4H	Sulfite impregnated—coniferous woods	1 to 5% Na_2SO_3	85 to 90	High	Poor	Excellent	Excellent[a]	Excellent blending pulp with slow stocks such as 4A, 4B, 4E, 4G—light weight
4J	Sulfite impregnated—hardwoods	1 to 5% Na_2SO_3	85 to 90	High	Poor	Fair	Good[a]	Too much power
4K	NSSC softwoods	To 12% Na_2SO_3 $NACO_4$	Under 85	Average	Good	Good	Good	Economics not good compared to above
4L	NSSC hardwoods	"	"	Average	Fair	Fair	Good	Same as 4K

[a] Sulfite and NSSC brighten the pulp as compared to groundwood from same wood.
Note: NaOH and steaming darken the pulps as compared to groundwood from same wood. Washing of cold soda pulps may be mandatory to provide economic sizing.

Pressafiner. In which case, the liquor is fortified for reuse. Our results indicate a little caustic goes a long way toward achieving the desired results.

Table I indicates results of cold soda processing of southern pine and oak for insulation board. The cold soda pretreatment and refining level were only two variables in the series of runs. Note the lowest caustic level on the pine produced one of the best boards and at the highest level NaOH consumption overtreatment resulted. It is also interesting that composite of $^2/_3$ cold soda pine and $^1/_3$ low freeness oak cold soda produced a good quality board. These results are representative only, and do not indicate optimum results possible with these woods by the cold soda method.

Difficulty in sizing without washing would be encountered on all pulps except perhaps ID. It should also be pointed out there is considerable change in pulp freeness due to washing, removal of solubles invariably drops the freeness (SFMC) of cold soda pulps.

Hot Sulfite Impregnated Wood

During investigation of various methods of producing insulation board stocks from both hardwoods and softwoods at Fiber Products Laboratory, hot sulfite impregnation was used and produced very unusual pulps. The pulps are bulky and lightweight with unusual resistance to beating.

The process is identical to cold soda except the impregnating chemical is a hot (250 to 300°F.) solution of sodium sulfite and water. The concentration of the solution depends upon the degree of treatment (usually 15 to 100 g.p.l.) actual chemical impregnated is generally 2 or 3% based on dry wood weight.

The sulfite has a brightening effect on the pulp and refined pulps are usually equal to or 2 or 3% G.E. brighter than the original wood.

The refined fibers are very strong, bulky, and very high freeness. It appears that it is uneconomical to refine to board machine freeness and furnish use will be as a mixture with abnormally slow, groundwood, cold soda, or steamed wood stocks. Such composites will produce boards with quality considerably above those being produced commercially today—lightweight, high strength, light in color.

It is doubtful that dense hardwoods will be used extensively by the hot sulfite method because of excessive power requirement and better pulp resulting from cold soda treatment. However, low density hardwoods and coniferous woods will probably be treated by this method for high quality, high strength decorative boards.

NEUTRAL SULFITE SEMICHEMICAL

This method is similar to hot sulfite impregnated pulps except sodium carbonate is used long with the sulfite and the chips are cooked in the liquor or steamed in the vapor phase following impregnation.

The NSSC process has been extensively applied for production of nine point corrugating medium and paper pulps, primarily on hardwoods.

These pulps appear to have little application in insulation board stocks due to low yield, higher cost, and the undesirable characteristics of high density in the board. This assumes pulps similar to nine point types at yields of, say, 80 to 85%. Extremely light treatments at higher yield levels are very similar to the hot sulfite impregnated pulps and will be considered in the same category in this paper.

GENERAL COMMENTS—PRODUCTION HIGH YIELD PULPS

If we consider a groundwood as produced from chips in a system equipped with hot water impregnation for using dry wood waste, all of the above high yield pulping systems utilize the same steps as follows:

1. Chipping
2. Chip preparation
   a. Impact milling for uniform sizing
   b. Chip washing for removal of tramp materials
   c. Chip screening
3. Digestion or impregnation
4. Refining
5. Pulp screening and/or cleaning

From this, we can see the only difference in production of all the high yield pulp types depends upon the chemical used, and, amount, degree of refining, and steaming. It is entirely conceivable that one system consisting of chipper,

Table V. Possibilities of High Yield Pulps for Use in Composites or Combinations to Produce Insulation Board Stocks

Slow stocks over 45 sec. SFMC freeness	*Free stocks under 20 sec. SFMC freeness* — *Coniferous groundwood from chips*	*Low density hardwood groundwood from chips*	*Coniferous steamed woods*	*Hardwood steamed wood*	*Cold soda softwoods*	*Cold soda hardwoods*	*Sulfite impregnated coniferous woods*	*NSSC coniferous woods and low density hardwoods*
Gdwd.—coniferous woods	Good	Good	Good	Fair	Good	Fair	Good	?
Gdwd.—low density hardwoods	Good	Good	Good	Fair	Good	Fair	Good	?
Gdwd.—high density hardwoods	Poor	Poor	Poor	Poor	Poor	Poor	Fair	Poor
Steamed wood—hardwoods	Good	Fair	Fair[a]	Fair	Good	Fair	Excellent	Good
Cold soda—hardwoods	Good[a]	Good	Good[a]	Fair	Good[a]	Fair	Excellent[a]	Good
Sulfite impregnated hardwoods	?	?	Poor	?	?	Fair	?	?
NSSC hardwoods	Good	Good	Good	?	?	Good	?	Fair

Code:
Excellent.....16 Plus U.S. Break Load.....500 plus MOR.....Light color
Good.....13–16 U.S. Break Load.....400–500 MOR.....Medium color
Fair.....11–13 U.S. Break Load.....300–400 MOR.....Dark
Poor.....Under 12 U.S. Break Load.....Under 300 MOR.....Very dark
?.....Unknown or never attempted, probably no good
Note: Above assumes approx. 50–50 or 1/3 slow–2/3 free stock in the composite.
[a] Indicates board shown in previous tables.

impact mill, chip washer, chip screen, Rapid, Cycle, M & D, or Grenco digester, and double disk refiners could produce any or all of the high pulp types thus providing the commercial operator with a very versatile system for the production of high quality board from all types of wood material and from all wood species economically available to the mill.

COMPARISON OF BOARD MAKING PROPERTIES OF HIGH YIELD PULPS

Above we have discussed the various high yield pulping methods for insulation board stock. There is an infinite variation in stocks these methods will produce with a wide variety of wood species.

Table II indicates results obtained using four of the high yield processes on southern gum. In this case, steamed wood and cold soda pulps produced the best boards. Since gum is a relatively high density wood, groundwood from chips resulted in a very poor pulp. These data very graphically demonstrate the drastic effect of light steaming and light caustic treatment. Hot sulfite while producing the lightest color of all the boards was very free and used considerable power. It is doubtful, if refined to machine freeness range, the added power would produce adequate commercial strength levels at economically feasible power cost.

Table III indicates results obtained using all the high yield methods for the production of insulation board stocks from southern pine and oak. Again, steamed wood and cold soda provided the best pulps from pine and oak. It is interesting also to compare the hot sulfite and NSSC pine where the latter dropped drastically in quality.

Also, note the very unusual quality of the pine hot sulfite pulp, very light and bulky with amazing strength when board weight is considered. Note, also, the excellent results produced when this type pulp is blended with slow stocks of oak, cold soda, and steamed wood. It is expected slow groundwood stock would produce similar results.

These data indicate the wide range of pulps that can be produced from these woods. The results are again indicative, but not optimum. Similar results could be expected with other coniferous and hardwoods using the same pulping methods. Actually, the woods used are not among the best pulping species and improved results would be obtained by use of more suitable woods.

Perhaps the most interesting portion of these data is the excellent board quality obtained by blending of free and slow stocks. In many cases, a superior board is produced from two stocks, neither of which will produce a commercially acceptable board.

Table IV provides a summary of the board making qualities of different types raw materials and processes. This information is based upon the data in the preceding tables, commercial operations, and a considerable volume of laboratory and semicommercial pilot plant work. This table contains a wealth of data, and it is suggested that the information be studied in detail.

Most of the information has been discussed previously, but the table provides a good summary and comparison of the various possibilities of high yield pulping methods for board stock.

COMPOSITE BOARD FURNISHES OF DIFFERENT HIGH YIELD PULPS

Multiple component board machine furnishes have been common to the board industry for years. Perhaps the most prominent use being the combining of bagasse and repulped waste paper. Other combinations are found throughout the industry. The main purpose of blending is to produce the widely diversified end-product quality requirements from one or two raw materials in a single or dual pulping system.

Table V indicates the results that could be expected from blends of all different type high yield pulps discussed previously. Some of the better blends were indicated in previous discussion and tabulated data.

In my opinion, the industry will undoubtedly trend toward wider use of composite board furnishes, and high yield pulps. The reasons are several. First, the wider product line and increased quality demands of the future will require pulp qualities beyond the capabilities of many present mill systems and processes.

Secondly, the changing raw material picture in the highly competitive wood resource field will require extensive use of cull species and wood waste products which, in turn, will require process changes to maintain end-product quality.

Insulation board and hardboard are in a very competitive market situation not only between the individual producers, but also because of alternate materials used for the same end application. Companies to remain competitive, must depend upon utilization of low cost raw materials (cull wood species and wood waste) and optimum processing. High yield pulps and composite boards would appear to provide part of the answer to future problems. Unfortunately, their effects upon transportation rates and labor costs will only be of a minor nature.

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Electrolytic Corrosion Protection of Paper Mill Equipment

T. R. B. WATSON

The paper presents a review of two electrolytic methods of controlling metallic corrosion: cathodic protection and anodic protection. Cathodic protection is universally accepted and widely practiced for the preservation of buried pipelines, water tanks, ships, etc., but it has found little application in the paper industry. Anodic protection is still being developed but it promises to provide a cheap and effective method of protecting carbon steel alkaline pulping digesters. Plant scale tests have been encouraging. A brief explanation is given of how each of the methods works, and the suggestion is made that cathodic protection should be given more consideration in design.

Corrosion is a purely electrolytic reaction and thus it would seem appropriate to fight it electrolytically. This paper will describe the principles of the two types of electrolytic protection using, respectively, cathodic and anodic control. Cathodic protection is a widely adopted, fully documented science of long standing: anodic protection is in its infancy and is still being explored, yet it promises infinitely more benefit to the paper industry.

CATHODIC PROTECTION

In cathodic protection, a small continuous electrolytic current is made to flow from a metal or graphite anode through an aqueous electrolyte to the surface of the metal to be protected. An electronic current of the same magnitude also flows from the anode to the protected metal through a metallic circuit. Although both electron and ion flow are essential to any electrolytic cell, it is proposed to explain how cathodic protection works by reference to the effect of the electron flow in the wires, rather than by the more usual explanation stressing the electrolytic part of the circuit. The story is made descriptive at the expense of strict technical accuracy, but the overall picture is true.

When a metal is reduced from its ore, the process uses up energy and this energy is retained by the metal until the day it corrodes back to an oxide again. In fact, it is this stored-up energy which powers the corrosion reaction and which makes metals "want to corrode." Again, it would seem fruitful to fight energy with energy.

When steel is immersed in water, iron atoms dissolve to become ions, leaving electrons behind in the body of the metal. These free electrons build up and the metal becomes more and more electronegative until the positively charged ions can no longer escape from the negatively charged steel. Thereafter, corrosion can only proceed if electrons are drained off in some way. The extent of the negative charge on the metal at this stage is called its "solution potential." It is a condition of equilibrium, wherein the stored-up energy in the metal or its "desire to corrode" is balanced by the degree of electrostatic charge.

Corrosion still continues, however, because electrons leak off and are removed from the steel by cathodic reactions involving combination with oxidizing agents or the evolution of hydrogen. These reactions may take place a mile, an inch, or a micron away from the corroding area. To prevent corrosion then, it is only necessary to supply to the metal more electrons than the natural cathodic reactions can use up. Take electrons away and it will corrode; put some in and it will stop. Make it a little more positive and it will rust; make it a little more negative and corrosion will cease. This is the principle of cathodic protection.

The solution potential of a metal depends upon the amount of energy it contains and this is the basis of the familiar galvanic series. Gold and platinum at one end contain very little energy, so they have little "desire" to corrode, while magnesium at the other end contains enough energy to balance a very high negative potential at equilibrium. Thus, if you were to couple gold or copper to steel in an electrolyte, it would drain electrons off and cause corrosion of the steel, but if you were to connect steel to magnesium the steel would be flooded with electrons and its corrosion would cease. Magnesium is widely used in this way to provide cathodic protection. Since in supplying electrons the magnesium is preferentially corroded and used up, the effect is often called sacrificial protection. The magnesium, in effect, corrodes instead of the steel. Zinc is also used as a galvanic anode and, in sea water, it has some advantages over magnesium.

Galvanic cells of this nature operate wherever two or more metals are in contact in an electrolyte. Older paper mill equipment often appears to be designed to incorporate as many metals as possible, each either corroding or protecting the other, but lately the widespread use of stainless steels and better recognition of galvanic relationships have greatly improved matters.

Another way of providing electrons is to "pump" them in via an applied d.c. voltage. The source of impressed current is usually a transformer-rectifier with the negative terminal connected to the protected equipment and the positive one feeding a "permanent" anode of graphite, high silicon cast iron, or platinum. These materials are not decomposed by the current and last indefinitely.

Cathodic protection got its start in the pipeline industry where, with millions of dollars tied up in thin steel pressure vessels, the industry just cannot tolerate corrosion. Today, every major pipeline on the continent is cathodically protected; so are the ships of the Canadian and United States navies and the cargo compartments of many tankers. Most people have a magnesium rod in their hot water tank at home. In the paper industry, where much of the equipment is immersed, cathodic protection would appear particularly appropriate but, surprisingly enough, this method has found little application. Instead, the industry has unanimously approached the problem from another tack: that of replacing anything that corrodes with stainless steel or some other noble alloy.

There is much justification for this viewpoint. Cathodic protection has important limitations. To be effective, anodes must be numerous enough and so disposed that the protected surfaces receive a fairly uniform current density. This means that you cannot, for instance, protect the inside of a pipe without having internal anodes over substantially the whole length. Anodes must not interfere with the function of the equipment and, since the flow of electrons to the metal must be balanced by a constant electrolytic current flow through the solution, cathodic protection will not work above the water line. Furthermore, rectifier installations and even galvanic anodes need occasional maintenance, if they are to keep going. Nevertheless, cathodic protection, where it is appropriate, will work very effectively and exceedingly cheaply and, as a tool, it should not be overlooked.

Magnesium anodes find good use in the protection of buried steel piping in the mill yard. For the most economical protection, newly laid pipelines should be coated and insulated off at both ends but existing bare pipes can be protected without digging them up. Even the steel hoops of buried wood stave pipelines may be preserved by the same methods, if they are bonded together. Cathodic protection of elevated water tanks is quite general and works well. An instance has been reported (*1*) of the successful protection of bronze groundwood decker facing wires, using magnesium anodes.

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ANODIC PROTECTION

So far, we have considered only the prevention of corrosion through control of the cathodic reactions, i.e., of supplying electrons faster than the cathodic reactions can use them up. This, of course, is only half the story for conditions at the anode also limit the corrosion rate. For attack to continue, dissolving metal ions must be able to react with the environment and thus escape. If this is prevented, corrosion stops. This is the principle of the resistance of the stainless steels which are covered by a "passive film" which shields the metal itself from its immediate surroundings. The exact nature of this film is highly controversial but it is agreed that it depends for maintenance and repair upon oxygen or some oxidizing reaction.

These reactions are intensified if the metal surface is made anodic and, if the environment is not sufficiently oxidizing, film repair can be supplied by electrolysis. In fact, if the metal alloy were made anodic enough, actual oxygen would bubble off at its surface but, in practice, only very little current is necessary. Sudbury, Riggs, and Shock (*2–4*) have shown that the resistance of stainless steels in borderline environments can be greatly improved by the passage of surprisingly small electrolytic currents and, furthermore, the throwing power is great, making the current far reaching. This points to the pleasant possibility of up-grading the cheaper stainless steels which, when supplemented with anodic protection, may be serviceable in environments now requiring more expensive materials.

In very strong oxidizing agents, such as concentrated nitric acid, even mild steel will develop a tenuous passive film and, in highly alkaline conditions, this film is more easily formed. Mueller (*5–7*) has shown that the interior surfaces of mild steel digesters used in alkaline pulping are passive for about half the cook. This means that 90% or more of digester corrosion takes place during the first hour or so, after which the metal "goes passive" and corrosion ceases. If this film could be maintained throughout the cook, digester corrosion would no longer be a problem. By passing an electrolytic current to stimulate formation and repair of the passive film, it has been demonstrated that this can be done.

The equipment required is very simple. Three flexible steel cables are suspended from the dome, and a high amperage, low voltage current is impressed between the cables and the digester shell. Special calomel electrodes are installed in several places in the shell and their indications together with the impressed voltage and current are continuously recorded. From these charts, the active or passive condition of the digester can be seen at a glance. Automatic control circuits vary the current to keep the digester surfaces in the passive range. Equipment is also provided to turn the power off, if the digester fails to passivate for any reason at the beginning of a cook.

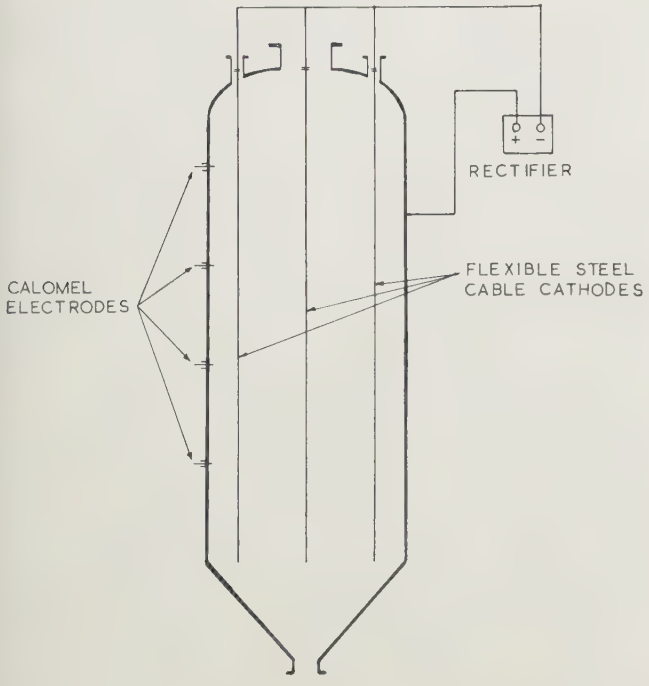

Fig. 1.

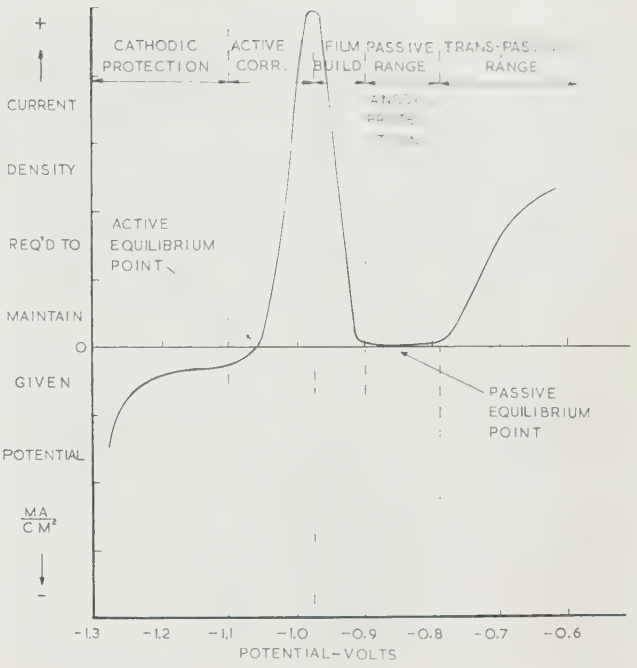

Fig. 2.

The polarization curve, published by Mueller, shows that at relatively cathodic potentials mild steel in cooking liquor does not corrode (cathodic protection). At the active equilibrium point, where no external current flows, corrosion proceeds rapidly and, as the potential is made more anodic, both the current required to maintain it and the corrosion increase exponentially. Suddenly, at about −0.95 v. (ref. calomel) the passive film starts to form, the resistance goes up and the current and corrosion rate drop sharply to practically zero. Throughout a wide range of potential thereafter, the passive condition is maintained and the metal is protected.

During a normal cook (with no current supplied), the digester ends up passive. After the blow, the incoming strong liquor boiling on the hot surfaces activates them and the vessel stays active for about an hour and a half, corroding strongly at the active equilibrium point. During the cook, the liquor becomes diluted, the temperature rises and the peak of the polarization curve drops. Suddenly, the digester shifts from the active to the passive equilibrium point and it stays there for the remainder of the cook. When anodic protection is applied, the current is turned on shortly after the digester is full, when most of the boiling action has ceased. A maximum current is required immediately to get the potential over the hump of the curve. It is pointed out that if the current is insufficient initially, it will merely increase corrosion. If all scanning electrodes show an immediate shift to the passive range, the current may then be greatly reduced, for the current density required to maintain the passive film is much less than that necessary to form it. The steel cable cathodes are so negative that they receive cathodic protection and thus do not corrode—a fortuitous circumstance. This method is presently in the development stage but plant scale trials have been most encouraging and have indicated that protection by this means can be had far more cheaply than by weld overlay.

CONCLUSIONS

Cathodic protection is a cheap and effective method of preventing corrosion of buried or immersed structures made of steel or other common metals. The principle has been largely overlooked by the pulp and paper industry but in design it would be well worth while to give this method consideration before resorting to more expensive materials.

The new science of anodic protection, though still being developed and expanded, promises to be of great value to the industry in the preservation of alkaline digesters and stainless steel equipment.

LITERATURE CITED

1. Duncan, R. D., *Pulp and Paper* 50, No. 3: 189–191 (1949); *Chem. Ab.* 43, No. 10, 4012 (May 25, 1949).
2. Sudbury, J. D., Riggs, O. L., and Shock, D. A., *Corrosion* 16, No. 2: 47t–54t (Feb., 1960).
3. Shock, D. A., Riggs, O. L., and Sudbury, J. D., *Corrosion* 16, No. 2: 55t–58t (Feb., 1960).
4. Riggs, O. L., Hutchison, M., and Conger, N. L., *Corrosion* 16, No. 2: 58t–62t (Feb., 1960).
5. Mueller, W. A., *Can. J. Technology* 34: 162 (1956).
6. Mueller, W. A., *Tappi* 40: 129 (1957).
7. Mueller, W. A., *Tappi* 42, No. 3: 179–184 (March, 1959).

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Cleaning and Filtering, and Fiber Recovering in Paper Mills

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This paper describes the operation of a vacuum disk filter in a cylinder board mill utilizing secondary fiber as a furnish. The machine was installed in conjunction with vortraps in a program for better cleaning of the filler stock. All the filler stock is passed over the filter subsequent to cleaning. Thus the filter is used both as a thickener for the filler stock and as a filter for the mill effluent.

Five years ago we were faced with the problem of an inadequate cleaning system of the filler stock of our cylinder paperboard machine. The type of cleaning required the removal of all types of heavy materials found in the low grade wastepapers we were using at the time.

The cleaning equipment we chose was the Nichols Engineering 10 in. Vortraps, which required consistencies in the range of 1/2 to 1% for efficient operations. This meant we would have to operate at considerably lower consistencies than we had been, and that our thickening capacity would have to be increased from 700 to 2500 g.p.m. It was our desire to obtain this additional thickening capacity without increasing our well water consumption, and if possible, decreasing the solids content of our mill effluent with an efficient saveall. The standard thickners available would not provide us with an effluent of low enough solids content without additional treatment. The cylinder type saveall required more floor space than was available. After investigating the field we believed that the machine which came closest to our requirements, that is thickening filtering and fiber recovery, in the allotted floor space was the Dorr Oliver vacuum disk filter.

Plans were then made to set up a boxboard filler system capable of handling 100 tons per day. The filler furnish is charged directly into a hydrapulper and extracted continuously at 3% consistency by a 2500 g.p.m. pump. At the suction side of this extraction pump dilution waters are added reducing the consistency of the stock to 0.075% in order to supply the Vortrap at an acceptable consistency. The stock is then thickened by the disk filter to 8 to 9% consistency. It is then diluted to a refining consistency in the discharge trough of the filter and pumped to a dump chest.

The purpose of this paper will be to explain the operation of the filter as a unit in the filler system, its effect on the overall operation of the mill and what it is doing for our mill effluent.

The author assumes that the construction and operation of a vacuum disk filter is familiar to most and needs little explanation. This filter, through the use of a split center shaft, produces two filtrates of different clarities. The first called the cloudy filtrate is drawn through the 60-40 mesh wire covering the disk then as the fiber mat is formed the second or clear filtrate is formed by drawing the water through the fiber and wire. Any variation of flow or freeness to the filter causes a change in the level in the vat. This is sensed by a pressure diaphragm in the bottom of the tub which through air controls, changes the shaft speed by a Reliance drive. This drive and control will maintain the level in the vat within an inch eliminating any need for manual control of the machine. Because of this we are able to maintain a uniform head by varying the speed regardless of the freeness of the stock, with no manual adjustment.

The aim of the system is to maintain a discharge to the creek of 9000 g.p.m. consisting of 650 gal. of clear filtrate and 250 gal. of bearing and condensor waters with a solids content of 0.1 lb. per 1000 gal. This is accomplished by adjusting the discharge ports of filter to deliver approximately 1200 gal. of clear filtrate with a solids content of 0.1 lb. per 1000 gal. and 1300 gal. of cloudy filtrate with a solids content of 3/4 lb. per 1000 gal.

The clear filtrate under ideal condition is the only white water discharged to the stream. This amounts to 650 gal. The remaining 550 gal. is used in the mill on the machine felt showers and the high pressure wash showers used for cleaning the wires on the disk filter after removal of the fiber mat.

The entire cloudy filtrate under ideal condition, is reused in the mill in the following manner: 335 g.p.m. are used for dilution on the discharge trough of filter, and 950 g.p.m. are returned to the suction side of the hydrapulper extraction pump as dilution water for the cleaning system. The additional 1000 gal. of dilution waters for the cleaning system are made up of machine white waters and well water.

What has this system done for us?

1. We have been able to reduce our consumption of fresh water to 900 g.p.m. or 10,000 gal. per ton. The largest saving along this line was the use of clear filtrate on our wet felt showers, which is approximately 400 g.p.m.
2. Reduced our fiber loss.
3. Provided us a uniform consistency on our filler system.
4. Reduced the solid contents of our mill effluent.
5. Provided adequate thickening area in a small space.

What are the operational and work problem?

The operational problems after an initial period of startup problems (which we will discuss later) have been very limited. The machine is checked by the beater engineer two to three times per shift and the level chart on the control gives immediate warning of any trouble. We have been able to run stocks of 400 to 600 S.R. freeness with no difficulty.

The original face wires of 30 × 40 type 316 stainless steel were replaced after approximately 5 years of service. Occasionally during that period we have lost one screen because of unusual wear. This type of failure is readily noticed by an increase in the solids content of the clear filtrate.

The original sectors as furnished were constructed with a mild steel backing plate which rusted out within the first year of operation. These plates were replaced with stainless steel and have operated satisfactorily ever since.

The filter is designed as a 12-disk unit, however, at the present time there are only 10 disks operating. We found that the narrow sluice next to the two end sectors would plug

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up with stock and overload the motor. Inasmuch as the filter turned out to have greater capacity than we needed, the simplest solution to this plugging problem was the removal of the two end disks.

The solids content of the mill effluent since the startup of this system has been reduced from 0.9 lb. per 1000 gal. to 0.4 lb. per 1000 gal. We feel that this can be reduced even more by the elimination of all discharge waters to the creek other than the clear filtrate which has a solids content of 0.1 lb. per 100 gal.

On the negative side there has been a somewhat higher B.O.D. which is in all probability attributable to the smaller effluent volume resulting from the tightening of the system. This system has not provided for any disposal of washup waters inasmuch as the filter is only in operation during production periods.

The system as it is now operating, has proved to more than have met the expectation of the mill personnel.

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Production Applications of Fourdrinier Foils

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Sheet formation may be improved by the substitution of foils for table rolls, if pressure ridges or high drainage rates produced by table rolls cause stock disturbances. The degree of formation improvement will largely depend on the percentage of table equipped with foils. Drainage capacity of practically all fourdriniers may be increased by the use of properly designed and dimensionally scaled foils. The use of foils, properly scaled and spaced generally results in closer wire supports than possible with rolls. Wire sag and associated problems are thus minimized. The use of foils eliminates problems of roll whip and vibration.

The various organizations and individuals who have pioneered research of fourdrinier foils deserve a vast amount of credit. Several excellent technical papers have been presented as the direct result of these extensive laboratory research programs.

However, production application of foils has been slow to be adapted within the paper industry. This may possibly be due to a need for correlation between laboratory and practical experiences.

The purpose of this paper, therefore, is to make available results and observations of a number of foil applications and to present conclusions reached from these applications. The experiences to be cited include machines operating at speeds varying from 100 to 2000 f.p.m. These machines were equipped with wires from 40 to 276 in. wide. Stock freenesses ranged from glassine to tissue. First, however, a general description of fourdrinier foils and an explanation of their functions may be helpful.

FOIL DESCRIPTION AND FUNCTIONS

A single foil has a visual similarity to a deflector or drainage board of the style used between table rolls. Both consist of a beam on which a replaceable wire supporting strip is mounted. The front edge of this strip is usually beveled to produce a scraping or doctoring operation on the underside of the wire.

The visual similarity ends at this point. Whereas the drainage board is given a totally flat top surface; the foil has a relatively narrow flat portion of its front top surface supporting the wire. The remainder of the upper foil surface is then made to diverge from the plane of wire travel.

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Both foils and deflectors are used to support the wire and to remove excess water from the underside of the wire. Spray and discharge from adjacent machine elements is also deflected away from the wire and into saveall pans.

A major and important difference between the two, however, is realized by the ability of a foil to develop a vacuum in somewhat the same manner as a table roll. Very basically, a film of water separates a moving wire and the flat frontal area of a stationary foil supporting this wire. This water film is in adhesion with both surfaces and tends to move with the wire. As it moves, its attraction to the upper foil surface causes the lower boundary layers of this film to follow the diverging surface of the foil. This vertical deflection of the film creates a vacuum which causes water from above the wire to drain through and maintain the film continuity.

A foil thus performs the doctoring and the deflecting functions of a drainage board plus the drainage producing functions of a table roll eliminating at the same time on of the major sources of sheet disruptions, typical of table roll operation.

Water is forced into the front nip of all rotating table rolls. A pressure area is formed at this point which forces water upward through the wire producing a pressure ridge which is readily apparent by inspection.

The beveled front edge of a foil effectively removes water from the underside of the wire. It, further, does not present an opening which would lead to the formation of a frontal pressure area and resultant pressure ridges. Sheet formation may, therefore, be improved when foils replace table rolls or combinations of table rolls and drainage boards.

Foil application decidedly differs from that of drainage boards. Placement of foils between rolls would produce additional drainage which would further flood the front roll nip of following rolls. This often would lead to formation of larger pressure ridges and intensified stock disruption.

Foils, consequently, are designed to be used in a series, totally replacing all table rolls and all drainage boards normally employed in that particular portion of the fourdrinier.

When installed in a consecutive manner, drainage produced by a leading foil is doctored from the wire and deflected to the saveall by the following foil. In addition to the virtual elimination of stock disturbing pressure ridges, foils may usually be spaced on closer intervals than required for suitable table rolls. Wire sag is, therefore, reduced and a greater area of wire is utilized for active drainage.

GENERAL FOIL TYPES

Foils may be of a fixed sectional profile machined from a solid bar of material. As it is often difficult to accurately forecast the optimum profile for a particular application, an obvious inherent disadvantage is frequently experienced with their use. Sectional profile appears to become increasingly critical with increases of machine speed and stock freeness.

Foils may also be designed to produce an adjustable profile. One existing patented design utilizes a two-piece construction. The front section has the usual front edge bevel and its upper surface provides a wire supporting flat surface. The rear section is hinged on this front strip and may be adjusted to diverge from the plane of wire travel at a variable angle.

A second existing design utilizes a normally flat topped shape which is capable of being uniformly deformed across its length to produce a cross section curve or camber. This camber is formed only in the middle portion of the cross section leaving a narrow flat width at both edges. The forward flat surface again supports the wire.

This design avoids an intersection between two hinged members. The camber also provides an easement from the wire supporting surface to the degree of divergence which produces maximum drainage efficiently. Beyond this point, the continuation of camber further provides a relatively open passage for the discharge of drained water.

These features are design attempts to form a smoother curve of developed vacuum which in turn inhibits shock and

disturbance to the top side stock. Gently curving foil surfaces may further minimize turbulence and stall, while the increased rear opening is believed to minimize kick-up as the drained water leaves the foil surface.

The particular foil applications to be described involved two basic foil designs. One style was of the adjustable camber type having a total $4^1/_4$ in. width of which approximately $^7/_8$ in. of front flat surface supported the wire. The other had a relatively narrow nonadjustable profile.

SIGNIFICANT APPLICATIONS

Most foil applications made and observed have been successful. The examples to be described, however, have demonstrated particular aspects of foil application, limitation, or capability.

One of the first applications made was on a glassine machine operating at 220 f.p.m. Four foils of the adjustable camber style were installed at $4^1/_2$ in. spacing which required the removal of five 3-in. diam. table rolls. These rolls had previously been spaced on $3^1/_2$-in. centers. Initial observations were encouraging; however, only a small portion of the diverging surface was effectively producing drainage. Foils having traverse serrations throughout this cambered midsection were later substituted to increase surface area in an attempt to extend the length of effective drainage. The improvement, if any, was exceedingly marginal. Drainage tests conducted with the use of collecting pans, however, revealed these foils to individually produce an average of 25% greater effective drainage than produced by adjacent table rolls.

The term, effective drainage, as used refers to the quantity of water actually collected. This distinction must be made as drainage produced by these slow turning table rolls undoubtedly exceeds the amount collected in the pans. Unfortunately, at slow speeds, centrifugal force developed is not sufficiently great to release all the drained water film from the roll surface. A large amount of this film is carried completely around slow turning rolls to form a substantial pool at their front nips. Some of this pool drains down the front face of such rolls in the form of irregularly spaced ropes. The remainder passes between these rolls and the wire and is forced back into the sheet as evidenced by the noticeable pressure ridge which is formed.

Since the actual drainage produced by each of these four foils was slightly different, the 25% increase in efficiency was approximate. For no presently explainable reason, the leading foil which followed a table roll produced the smallest amount of drainage. The other three units produced very comparable amounts of drainage. Variations of camber adjustment and consequently foil shape had very little effect on the amounts of drainage produced. This led to the premature conclusion that foil shape was not particularly important. However, later trials on other machines have proved foil profile to become progressively critical as speeds increase and stocks become freer.

Although the performance of these particular foils was encouraging, in reality, little drainage benefit was achieved. The five replaced table rolls had previously produced five average units of drainage. Four foils, supporting an equal length of wire and averaging 25% more drainage per unit, again produced five average units of drainage. Obviously, no increase of total drainage resulted. No appreciable effect was apparent in the formation of the sheet produced. Although pressure ridges were not formed over these foils, 25 rolls and 25 pressure ridges still remained.

Observation had shown the width of these foils to be far larger than necessary for this application involving slow stock at slow speed. Consequently, a series of six narrow fixed profile foils were assembled on an open grid support and installed directly forward of the suction boxes of this same machine. Each foil had an $1^1/_8$ in. total width with a $^3/_8$ in. wide wire supporting flat at the front followed by a surface diverging from this plane on a 4 in. radius. These were mounted on $1^3/_8$ in. spacing on the supporting grid. The resulting assembly somewhat resembled a slotted suction box cover having an 8 in. width, five slots, and contoured land surfaces. The last two table rolls of this fourdrinier were removed to permit installation of the foil assembly.

The six foils produced three times the effective drainage as had previously been produced by the two replaced rolls. As this occurred in the same length of wire previously occupied by rolls, this foil design appreciably raised the drainage capacity of this machine.

A second and somewhat unexpected benefit was also realized. Wet streaks previously noticeable over the suction boxes virtually disappeared.

Both foils and rolls operate most efficiently under wet conditions. In slow applications, film carry around and subsequent flooding of roll front nips tends to inhibit drainage selectivity. In addition, the ropelike front side drainage produced by such rolls may further influence formation of wet streaks.

Foils have no rotation and naturally no carry-around. The drainage efficiency of any point on a foil surface, therefore, reflects the amount of water directly above that point. The drainage produced by foils is, therefore, greater at wet streak areas than at relatively dry adjacent areas. In effect, a moisture leveling operation is performed which increases the uniformity of moisture level across the fourdrinier width.

As a suction box naturally pulls more air, and with it moisture, through the drier and more porous areas of a mat, it is frequently necessary to resort to high vacuums to remove wet streaks showing over these boxes. Increased porosity and pinholing of the sheet may result. The leveling effect of foils placed directly in front of suction boxes reduces wet streaking and minimizes pinholing dangers. Foils of this same narrow fixed shape have since been installed on other machines with very similar results. These successful installations have included applications on glassine, typewriter carbon, saturating, towelling, and tissue grades with operating speeds from 200 to 600 f.p.m.

Another early application was made on a machine producing a number of magazine grades at speeds ranging from 200 to 600 f.p.m. Here two adjustable camber foils were installed directly after the breast roll. Space limitations and inaccessibility did not permit collection and measurement of drainage by the use of pans. Real operating benefits were realized, however, which indicated that drainage was increased and disruption forces reduced.

Forty feet per minute machine speed increase was realized in the production of a 30-lb. sheet. While no speed-up was attempted while running 90-lb. cover stock, a substantially better formed sheet was produced. This may be partially due to absence of pressure ridges at this critical forming area. Increased drainage capacity, however, permitted the lowering of headbox consistency when producing the heavy sheet. This also is a probable factor influencing the formation improvement achieved.

Installation of two adjustable camber foils of the same design was made on a groundwood specialty machine normally operating at 950 f.p.m. This brought forth a better understanding of the previously mentioned transverse serrations provided on the midsection area of these foils.

These serrations had not produced any appreciable effect at two hundred odd feet per minute. At 950 f.p.m., they produced unexpectedly little drainage. Inspection showed the continuity of the water film between the wire and foil surface to be broken at the vicinity of the leading serrations. This condition remained regardless of camber setting.

Serrations, and presumably any other surface irregularities, of foil surface apparently produce increasing turbulence at higher speeds. This, in turn, prematurely ruptures the film, preventing development of vacuum which results in loss of foil drainage efficiency.

Smooth surfaced foils later installed on this same groundwood machine produced satisfactory results.

Drainage samples collected from smooth surface foils showed effective drainage to be equal to that collected from the 6 in. diam. table rolls normally used on this groundwood machine.

Table rolls spacing on this machine is on 9 in. centers and cannot be materially reduced. The two foils evaluated, however, were installed on 6 in. spacing which actually could have been further reduced.

Here, the equal drainage produced by foils in combination with closer space results in better utilization of the fourdrinier wire for drainage purposes. In this particular application, installation of a full table of foils could conceivably produce upwards to 50% more total drainage capacity.

Two additional installations of serrated foils made about the time of the original installation on the above groundwood machine were primarily notable due to their failure. One involved a book machine operating at 1500 f.p.m. while the other was made on a newsprint machine operating at 2000 f.p.m. Again serrated foils produced only limited drainage. Circumstances have not yet permitted evaluation of smooth surfaced foils in either instance.

Installation of two smooth surfaced adjustable foils have been made, however, on a second 2000 f.p.m. newsprint machine. Mill personnel are currently conducting a series of drainage tests, placing these two units in various locations on the fourdrinier table. Considerable enthusiasm has been expressed although performance data obtained have not been released by the mill.

A foil installation on a 2000 f.p.m. tissue machine has not yet proved satisfactory. The large degree of drainage produced caused holes to be formed in the sheet. It must be remembered, however, that solid table rolls produce even more disastrous results if used under similar conditions.

Some means of reducing foil drainage efficiency without creating turbulence must be incorporated in foil design to produce satisfactory results in high-speed, free stock applications. Grooving of foil surface in the direction of wire travel may be a possible solution which will permit controlled drainage with minimum disturbance.

At present, however, it appears that properly designed foils may be used to advantage in most applications normally utilizing table rolls.

SUMMARY

The application of foils to operating fourdriniers may be summarized into a surprisingly few statements.

1. Foils should be applied only where specific problems exist. Foil design and location may then be logically applied to achieve desired results.

2. No less than two foils in sequence should be used for evaulation purposes. Drainage occurring at the rear of the first foil plus that achieved by the front of the second measures the drainage efficiency of the first unit.

3. The last foil of a series must be followed by a drainage board to prevent excessive flooding of the front of a following roll.

4. Drainage efficiencies of foils should not be compared directly to that of table rolls. Relative practical spacing of foils and rolls must also be considered to determine the drainage achieved through a unit length of wire.

5. Foils may improve formation by (a) elimination of table roll pressure ridges and (b) increased fourdrinier drainage capacity may permit reduction of headbox consistency.

6. Relative effective drainage produced by foils directly compared to rolls varies with machine speed. (a) individual foils may produce the greater effective drainage at slow speeds and (b) individual rolls generally produce the greater effective drainage at high speed.

7. Smooth surfaced foils create less turbulence and produce greater drainage than rough surfaced foils.

8. Adjustability of surface divergence becomes progressively more critical with increase of machine speed and stock freeness.

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Note: Since presentation of this paper, improvement of foil design has been made. Improved dimensional control and surface finish has greatly improved operating results. Approximately twenty-five new style foils have been placed in service during the period from June 1, 1961 to date. Many of these have replaced the original foils cited in the above paper. Where direct comparison has been possible, it is apparent that drainage rates have been roughly doubled over those obtained using the foils described in the above paper. Consequently this paper is out dated and should either be so preferenced or discarded.

Reducing Down Time on a Recovery Unit

C. H. JOHNSON, C. P. ELMORE, AND R. N. BRUCE

With the increased importance of the recovery unit in a pulp mill, there is increased emphasis for proper utilization of scheduled outages of the recovery unit. Planning and organizing for shutdowns is an integral and necessary function of both the operation and the maintenance departments. Neat and accurate operating records, equipment history, and an indication of the results of the work accomplished during previous outages should be available to the people planning the next shutdown. Sufficient time must be allowed prior to any shutown for the procurement of manpower and materials to accomplish the necessary work during the outage. A thorough inspection by a qualified person will point out any areas of potential trouble. Both internal and external inspection should be made with particular attention given to areas of possible corrosion, erosion, or overheating. The results of this inspection are the primary source of information for planning the next outage. Proper maintenance plus preventative maintenance is necessary to provide the trouble free continuous service required of a recovery boiler.

The importance of the heat and chemical recovery unit to a pulp and paper mill is increasing as mills grow and as large recovery units are required to handle larger percentages of the mill's recovery capacity. In recent years the majority of new mills have been built with a single recovery unit to process all of the chemicals from the pulping operation. Spare or standby recovery capacity is a thing of the past.

As the importance of the recovery unit increases, so also the utilization of downtime becomes more critical. With the advancement to higher pressures and temperatures, recovery boilers must be fired and maintained more intelligently if continuity of service is to be achieved. It is the purpose of this paper to assist the operation and the maintenance organization in obtaining the maximum benefit from scheduled outages and to reduce to a minimum costly unscheduled shutdowns.

Maintenance of the recovery unit is integrally associated with the operation, poor and haphazard operating techniques result in more downtime and extremely high maintenance costs. More and more organizations are realizing that overloading existing recovery capacity is extremely costly, both from steaming efficiency and increased maintenance standpoints. That industry is more conservative in its selection of recovery capacity is evidence of this.

Overloading taxes all portions of the recovery system and increased maintenance to pumps, fans, motors, smelt spouts, refractories, and other components can be expected. Load swings should be made as smoothly as possible to prevent

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upsets in firing conditions, which also affect maintenance requirements.

Operating records can be a great assistance in indicating areas of potential trouble, but the records must be complete and accurate. Operators must be trained on a day-to-day basis to keep proper records. The logs should be kept on the basis of what is read, not on what the reading should be or were in the past. Differences between present and past performance should be noted and the reasons for the differences ascertained. Proper observation and reporting of the differences in performance can prevent costly repairs. As an example, an undiscovered tube leak can cut many adjacent tubes in a short period of time, particularly on high pressure units. These added repairs can be eliminated by quick action if the immediate trouble is properly determined.

The training of operators by supervisory personnel must be a continuous process. When a change in operation is made, the reasons for it should be made plain to the operator so that he will know why as well as how the job is to be performed. The field service engineer can greatly assist in the training of plant personnel. The operators should look on him as an aid to them and not just as a visiting expert there only to criticize. These servicemen are available for training at each visit and their knowledge should be used to advantage at each opportunity.

To utilize fully scheduled outage time, complete planning is not just desirable but necessary. Meetings between maintenance and operating personnel are required to properly coordinate an outage. Complete communications are necessary to insure the availability of both the equipment to be repaired or changed and the manpower and material required to accomplish the desired work.

These meetings should not be left to the last minute but should precede an outage by sufficient time to allow for the procurement of materials and scheduling of maintenance forces to accomplish the specified work.

The operation department should set the schedule of the outage to coincide with the maintenance requirements. Quite often startup and shutdown procedures can be accelerated to provide more working time. Proper understanding of the best operating techniques and scheduling of the component steps will allow for accelerated startups and shutdowns. Consult the boiler manufacturer for direct recommendations in this matter.

When the unit is cold, cleaning must be done by hand and is subject to the limitations of the personnel who are working inside the boiler. Therefore, any cleaning during the shutdown procedure reduces the time required to hand clean the unit. When liquor is removed from the furnace and auxiliary fuel is being fired to burn out the bed, a visual inspection of the convection passes will point out any areas of concentrated buildup. This buildup can be removed easily be sootblowing or hand lancing. Auxiliary firing produces lower gas temperatures in the convection passes. This lower temperature changes the characteristics of the deposit, permitting easier removal by external forces, such as sootblowers. Also, this is an opportune time to boil out and clean up the direct contact evaporator, whether cascade or cyclone. Check the pH of the flush water to insure that a high pH is maintained during this period. This pH control is particularly important if oil is used as the auxiliary fuel. If uncontrolled, the pH can drop to a value of two or three in a very short time and cause acid corrosion in the wetted areas. Caustic soda may be added to control the pH.

To speed up repairs, it is highly advisable to have the required materials available at the job, not in the warehouse. This can be arranged several days before the outage. Also, a complete set of miscellaneous parts should be on the job. Having two or three mechanics wait for a helper to go to the warehouse for a bolt or other small component is a completely needless waste of time and money. Having proper materials on the job will eliminate this waste. Rigging, welding machines, and cables can be set before the outage, thereby reducing the waste of outage time.

Also, arrangements should be made to have sufficient and proper manpower available for the repair work planned. At times it is desirable to have the boiler manufacturer or contractor bring in a work force to accomplish a major repair. This outside contractor can hire men on a daily basis, thereby relieving the mill of the fringe cost of hiring a small work force for a short time.

At each outage a complete inspection should be made by a qualified person who is thoroughly familiar with the unit from the standpoint of both operation and maintenance. This same individual should make the inspection at each outage. This comparative type inspection will often point out areas of trouble which cannot be noticed at any one time. A check list for the inspection is helpful in assuring that all areas have been examined, this is a must for any degree of preventative maintenance.

The inspection should cover all components of the unit. Micrometer readings of the superheater tube diameters should be taken to point out any metal loss. Complete records of the tube diameters and locations of the measurements clearly show areas of possible trouble. Visual inspection of cleaned tubes will disclose blisters, erosion (near sootblowers and rodding ports), and corrosion (economizer tubes). Rubbing a tube by hand will often reveal imperfections which cannot be determined visually.

It is important that an inspection of the furnace bottom, refractories, tube fins, and seals be made during every outage, and necessary repairs made to prevent unscheduled outages due to smelt leaks.

The internals should be inspected at least once a year. The drums and headers should be inspected for cleanliness and checked against the previous inspections. The drum internals should be tight and in place. Auxiliary firing equipment, safety interlocks, and emergency devices should be checked and kept in good operating condition.

Flues, ducts, fans, and evaporators should be inspected for corrosion. Dampers should be checked for operation and possible distortion and the steam coil air heater for cleanliness. Proper feedwater treatment and close control thereof is one of the major steps that can be taken for the prevention of superheater tube failure due to carryover; also as prevention of possible boiler and furnace tube failure due to scale formation.

Technical assistance is available for inspections. Service engineers from the manufacturers along with chemists or corrosion engineers can be of great assistance for specific problems.

The following shutdown should be organized on the basis of this inspection. Work items should be broken down into those which must be accomplished and those which it would be desirable to complete. Materials should be ordered at this time, including materials for doubtful items.

Prior planning and advanced ordering is necessary because boiler manufacturers cannot stock spare parts. They must be fabricated for each individual order, which means that shipment of spare parts will normally require an extended period of time, depending on the nature of the item.

Material history records can be of great assistance in planning for shutdowns and can, at times, prevent costly unscheduled outages if properly used to institute preventative maintenance. Also every item that can be repaired while the unit is in operation should be attended to. All equipment whether boiler proper or auxiliary should be investigated to see if it falls into this category.

In summary, valuable outage time should be utilized to a maximum. Prior planning and complete coordination of efforts of the maintenance and operating groups will provide the trouble free service required of a recovery boiler.

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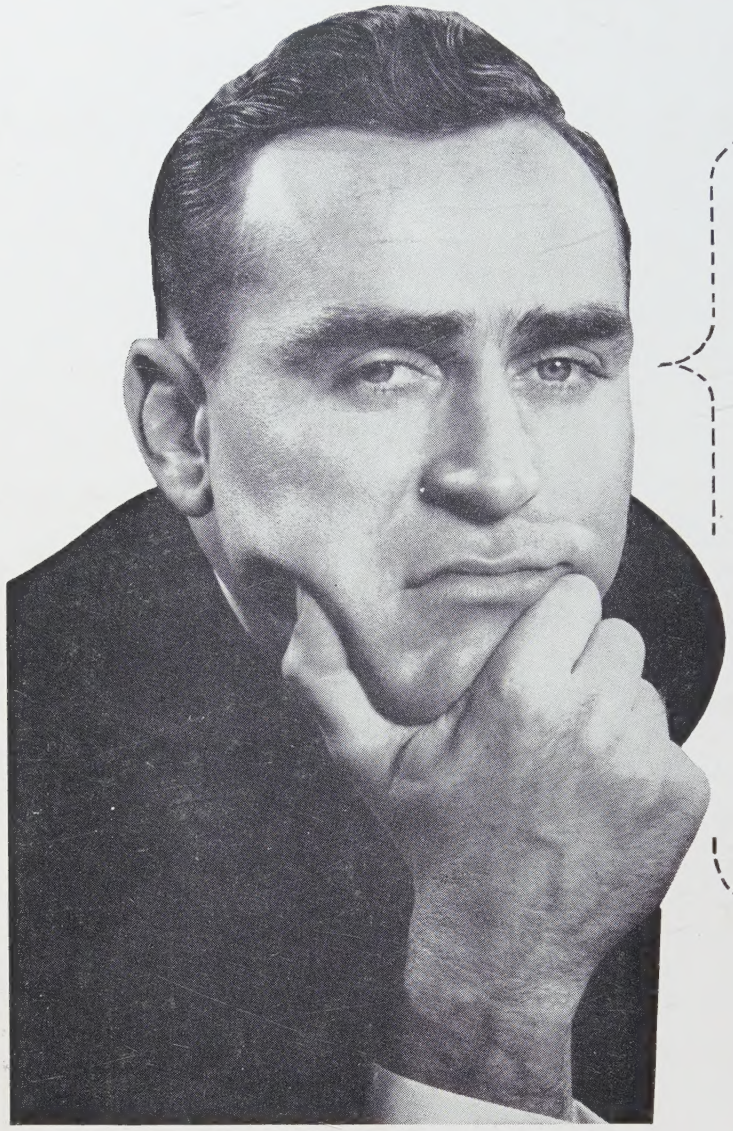
Thinking of coating or filling with clay?



Should we use a trailing blade... or an air knife?

Maybe it should be a roll coater... or a reverse roll?

What about a size press?

Double coating? Simultaneous or successive?

On or off the machine?

Wet or dry storage?

Fill at the secondary headbox?

Best place to furnish the clay?

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